

# SOIL SCIENCE

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## SOIL SCIENCE

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## ERRATA

- Page 204. The figures in table 4 should be multiplied by 2 to convert them from parts per million of nitrates in the solution, to parts per million in the dry soil. This correction should be applied also to the figures in the paragraph next to the last on page 205.
- Page 413. Title of paper should read: "The Colorimetric Determination of Soil *Nitrates* in a Colored Water Extract."

## NITROGEN LOSSES UNDER INTENSIVE CROPPING<sup>1</sup>

J. G. LIPMAN AND A. W. BLAIR<sup>2</sup>

*New Jersey Agricultural Experiment Station*

Received for publication February 5, 1921

In an earlier paper attention has been called to the loss of nitrogen and carbon in cultivated land as compared with the loss on land abandoned to grass and weeds. The work covers a period of 9 years, and deals with a loam soil (inclining to a sandy or gravelly loam) which during the period of the experiment received only moderate applications of a nitrogenous fertilizer. A partial bibliography was included.

The work reported in this paper covers a period of 20 years and deals with a loam soil which, with certain exceptions to be noted later, has been heavily fertilized and manured regularly throughout the 20 years.

As originally planned the experiment included a study of the availability of nitrogen in different materials, as measured by the amount of nitrogen won back through the crop, the percentage of nitrogen in the crop as affected by the treatment, denitrification studies, and the effect of the various treatments on the income and outgo of nitrogen in the soil.

Fairly complete reports of this work have been published from time to time (4, 5), but these do not give the results of the nitrogen determinations in the soil later than 1912. It is proposed in this paper to consider particularly the nitrogen income and outgo of the soil.

The work has been carried out by means of cylinders which have already been described (4). The arrangement of these cylinders and the plan of fertilizer and manure treatment are shown in figure 1. The cylinders were installed and the first crop grown in 1898.

A 5-year rotation consisting of corn, oats (2 years), wheat and timothy has been carried out on the cylinders. Four such 5-year periods were completed with the timothy crop of 1917.

Two residual crops (a crop immediately following the main crop) have been grown during each 5-year period, but the fertilizers and manure are always applied for the first crop.

Cow manure was used at the rate of 16 tons per acre; nitrate of soda at the rate of 160 and 320 pounds per acre; dried blood and ammonium sulfate were used in amounts equivalent to 320 pounds of nitrate of soda per acre.

<sup>1</sup>Paper No. 23 of the Journal Series, New Jersey Agricultural Experiment Stations, Department of Soil Chemistry and Bacteriology.

<sup>2</sup>The authors are indebted to Dr. H. C. McLean, formerly Research Chemist, and Mr. A. L. Prince, Assistant Chemist, for a large part of the analytical work reported in this paper.



Series.	<i>C</i>	<i>B</i>	<i>A</i>
1. Check.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
2. Minerals.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
3. Minerals, manure, solid, fresh.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
4. Minerals, manure, solid and liquid, fresh.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
5. Minerals, manure, solid, leached.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
6. Minerals, manure, solid and liquid, leached.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
7. Minerals, nitrate of soda, 5 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
8. Minerals, nitrate of soda, 10 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
9. Minerals, manure, solid, fresh; nitrate, 5 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
10. Minerals, manure, solid, fresh; nitrate, 10 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
11. Minerals, manure, solid and liquid, fresh; nitrate, 5 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
12. Minerals, manure, solid and liquid, fresh; nitrate, 10 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
13. Minerals, manure, solid, leached; nitrate, 5 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
14. Minerals, manure, solid, leached; nitrate, 10 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
15. Minerals, manure, solid and liquid, leached; nitrate, 5 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
16. Minerals, manure, solid and liquid, leached; nitrate, 10 gms.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
17. Minerals, sulphate of ammonia, equivalent to 10 gms. nitrate.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
18. Minerals, dried blood, equivalent to 10 gms. nitrate.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
19. Minerals, manure, solid, leached; sulphate of ammonia as in 17.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>
20. Minerals, manure, solid, leached; dried blood as in 18.	<input type="radio"/>	<input type="radio"/>	<input type="radio"/>

FIG. 1. DIAGRAM OF EXPERIMENT

All cylinders except the three of series 1 have received annual applications of acid phosphate at the rate of 640 pounds, and muriate of potash at the rate of 320 pounds per acre. Experience has shown that such applications furnish an excess of phosphoric acid and potash and thus make it unnecessary to take into consideration the amount of these constituents contained in the manure.

In certain cases the nitrogenous fertilizers were used alone and in others they were used in combination with the manure. The applications of manure are unusually heavy for general farm practice, but this was a part of the plan for denitrification studies.

When the soils were first prepared and placed in the cylinders a liberal application of lime was made to all, but no lime was again applied for 10 years. Thus for the first 10 years the treatment for the three cylinders (treatments in triplicate) of any given series was the same, and therefore, the results for each series of three cylinders could be averaged. In the spring of 1903—beginning of the second 5-year period—the soils of each series were removed and thoroughly mixed in one heap in order that slight differences might be equalized, and then one-third of this mixture returned to each of the three cylinders constituting a series. This procedure was repeated in the spring of 1908, that is, after 10 years of cropping. At this time there was placed in each cylinder the equivalent of 174.1 pounds of air-dry soil. Up to this time the treatment for the three cylinders of a series had been kept uniform. From this time forward a distinction is to be made. Those cylinders designated as *A* are to remain continuously unlimed; those designated as *B* and *C* are to be limed once every five years, and the *C*'s, in addition, are to grow two green-manure crops (legume) during the 5-year period.

These green-manure crops follow the corn crop and the first residual crop. In accordance with this plan the *B* and *C* cylinders received 125 gm. of pulverized limestone each, in the spring of 1908. Later this application was considered insufficient and the treatment was repeated in 1909. Aside from the lime and green manure, the three cylinders of a series receive like treatment.

Beginning in 1908 each season has given 60 distinct crops (one for each of the 60 cylinders), or a total of 600 crops covering the period 1908 to 1917, inclusive. To this number must be added 60 residual crops for each of the two 5-year periods, or a total of 720 crops for the 10 years.

A careful record has been kept of the amount of fertilizer and manure applied to each cylinder, and also of the dry matter in the form of crops removed from each. It is thus possible to calculate how much nitrogen has been applied during the 20 years and how much has been removed in the form of crops.

Nitrogen was determined in the original soil, and determinations have since been made on samples collected at intervals of about 5 years. It is thus possible to give a fairly complete inventory of the nitrogen income and outgo for the 20-year period.

As pointed out in an earlier paper (5) there has been a loss of about 40 to 70 per cent of the applied nitrogen as computed by taking the difference between the total amount applied and the amount won back through the crops.

Such losses may occur through the loss of nitrogen compounds in the drainage waters and also through the escape of gaseous nitrogen and ammonia into the air. Our knowledge of the proportions which are lost through one of these channels as compared with the other is limited. The problem of getting at the amount of loss through the drainage waters is not such a difficult one, and has already been studied by a number of investigators in this country and abroad, but a determination of the relative amount of nitrogen lost as nitrogen gas and as ammonia is a more difficult proposition.

In the work reported here no attempt has been made to measure *directly* the nitrogen that is lost through the channels mentioned, but the subject is considered from the standpoint of nitrogen gains or losses as indicated by the analysis of the soil and of the crops that are taken off, and the original nitrogen content of the soil plus the nitrogen applied in fertilizers and manure.

#### LOSS OF NITROGEN FOR THE FIRST TEN-YEAR PERIOD

The average results for the first 10 years (first and second rotations) are shown in table 1. This table shows the amount of nitrogen that was originally present in the soil, the amount that was applied to each cylinder during the 10 years, the amount that was removed by the crops and finally the total loss exclusive of the amount removed by crops.

The greatest loss was 39.38 gm. from series 12 and the least 25.12 gm. from series 19, with an average of 32.24 gm. It may be pointed out that the loss was almost as great from those cylinders that receive large applications as from those that receive small applications of nitrogen. It is generally true however, that there was less nitrogen removed in the crops from those cylinders that receive light applications than from those that receive heavy applications.

The average loss—32.24 gm. per cylinder—is equivalent to 1032 pounds per acre if the entire depth of the soil (about 10 inches) is included.

This means an average loss of 103 pounds of nitrogen per acre annually. This is an unusually heavy loss, but it must be remembered that the treatment to which the soils were subjected also was unusual to the extent that they were shoveled over more than would occur under field conditions and thus oxidation processes were accelerated.

It may also be pointed out that from soils rich in nitrogen, the loss is great. It is well known that there is a tendency toward equilibrium in soils. A poor soil left to itself may gradually increase its nitrogen content while a rich soil that is under constant tillage is very likely to decrease.

Discussing this tendency to equilibrium, Warington (9) says:

We have spoken earlier in this lecture of the natural limits to the accumulation of nitrogen in the soil: we can now, I think, perceive some of the causes of such limits.

The addition of organic matter to a soil either as crop or weed residue, or as farmyard manure, at once makes that soil a suitable home for the animal life, the fungi, and the bacteria whose function it is to reduce organic matter to the condition of inorganic matter. An increase of organic plant residue or manure thus creates some of the conditions favorable to its own destruction. The rate of oxidation in the soil is now no longer what it was; the oxidizing agents have increased with the material to be oxidized. If, therefore, a soil is laid

TABLE 1  
*Loss of nitrogen from cylinder soils during the first ten years—Average of sections A, B, and C*

SERIES	a PRESENT 1898	b APPLIED 1898-1907	m (a + b) TOTAL PRE- SENT AND APPLIED	c REMOVED 1898-1907	d PRESENT 1907	n (c + d) TOTAL RE- MOVED AND PRESENT	m - n LOSS EX- CLUSIVE OF CROPS 1898-1907
	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	155.47	0.00	155.47	11.213	108.70	119.91	35.56
2	155.47	0.00	155.47	12.966	111.23	124.20	31.27
3	155.47	40.22	195.69	21.877	137.78	159.66	36.03
4	155.47	42.76	198.23	26.982	132.17	159.15	39.08
5	155.47	38.25	193.72	22.042	142.44	164.48	29.24
6	155.47	42.17	197.64	24.072	144.57	168.64	29.00
7	155.47	8.58	164.05	17.865	116.84	134.70	29.35
8	155.47	15.50	170.97	22.371	116.52	138.89	32.08
9	155.47	48.85	204.32	26.469	138.64	165.11	39.21
10	155.47	55.77	211.24	32.140	141.80	173.94	37.30
11	155.47	51.96	207.43	33.136	138.17	171.31	36.12
12	155.47	58.31	213.78	36.698	137.70	174.40	39.38
13	155.47	46.83	202.30	25.836	150.10	175.94	26.36
14	155.47	53.80	209.27	30.297	149.86	180.16	29.11
15	155.47	50.75	206.22	26.964	147.50	174.46	31.76
16	155.47	57.72	213.19	32.457	147.97	180.43	32.76
17	155.47	16.13	171.60	19.584	124.58	144.16	27.44
18	155.47	15.60	171.07	18.736	123.40	142.14	28.93
19	155.47	54.31	209.78	28.004	156.66	184.66	25.12
20	155.47	53.83	209.30	26.777	152.86	179.64	29.66
Average . . . .	155.47	41.74*	193.04	24.824	135.97	160.80	32.24

\* Average of 18.

down in pasture or receives an annual dressing of farmyard manure, the nitrogen in that soil will only increase so long as the annual increment of organic matter exceeds the annual decrement by oxidation. If this increment is a limited quantity it will be met before long with an army of destroyers competent to effect its destruction. The richest soils are thus the most liable to waste and demand the greatest exercise of the farmer's skill to preserve their condition.

When the conditions of the soil are changed, when the pasture is plowed up or the arable land is left without manure, there is at first a rapid loss of soil nitrogen, but the rate of loss soon diminishes. The organic matter most easily attacked has disappeared. The army of

oxidizing organisms has been reduced by starvation. A partial equilibrium is established when the annual destruction of organic matter amounts to little more than the annual residue of crop and weeds; but an absolute equilibrium is reached only when the annual loss of nitrogen is equaled by the atmospheric supply. In every case nature seeks to establish an equilibrium.

Russell (7) expresses about the same thought when he says:

Unfortunately on our present knowledge it is impossible to maintain a high content of nitrogen on cultivated land except at a wasteful expenditure of nitrogenous manure.

In discussing the loss of nitrogen from Rothamsted soils during a period of 50 years of cropping, Dyer (3) cites an annual balance of nitrogen unaccounted for amounting to nearly 70 pounds per acre on plots that received annually applications of ammonium salts equivalent to 86 pounds of nitrogen per acre. Dyer believed that this loss was largely in the form of nitrates that passed into the drains. He says:

Actual analyses of the drainage waters running from the pipes showed quantities of nitrates which go far toward accounting for the lost nitrogen, but the drain pipes only discharge a fraction of the total drainage, and a large quantity of drainage must often find its way downward below the level of the drainpipes when the pipes are not running, and even when they are.

Most, if not all, of the loss on the chemically manured plats may therefore, be said to be in the form of nitrates. It is possible that under certain conditions of weather there may be slight loss by evolution of free nitrogen from the crop residues of the surface soil on these plats but there is no evidence to show that any of the nitrogen added in the form of chemical manures is thus lost. But fermentative decomposition involving evolution of free nitrogen no doubt takes place on the heavily and continuously dunged plats, with their high quantity of organic matter and in a less degree on the rape-cake plat. It is to be borne in mind, however, that the quantity of dung used in these continuous wheat growing experiments is, on the yearly average, far less than would be used in practical agriculture on any of the rotation systems.

With reference to the exhaustion of the nitrogen supply of soils under continuous wheat growing, Dyer (3) says:

For the wheat crop has completed its growth and is harvested long before nitrification has ceased in the soil, and there is no crop to take up either the balance of the nitrates left unused by the wheat or the larger balance formed after the active growth of the wheat is over; and these nitrates must pass away in subsoil drainage during the winter, except when the rainfall is small, or in those latitudes exposed to long winter frosts. This drainage, even on unmanured land, involves a serious loss of nitrogen, and a still greater loss when the yield of the crops is artificially increased by the use of nitrogenous manure.

Snyder (8) has called attention to the enormous losses of humus and nitrogen which take place in continuous wheat growing in Minnesota. He writes:

One of the chief causes of the decline of fertility of old grain soils, is this loss of nitrogen by the rapid decay of the animal and vegetable matter of the soil, which has been accelerated by the continuous cultivation of grain. Thus of the 1700 pounds of nitrogen lost during the eight years of continuous wheat cultivation, 300 pounds only have been utilized by the wheat

as plant-food and 1400 pounds have been lost by the decay of the humus and the conversion of the nitrogen from the insoluble and stable humic forms to gaseous and soluble forms which are readily lost.

When wheat was grown continuously there was an annual loss of over 2000 pounds per acre of humus, due to the fermentation and decay of the animal and vegetable matter of the soil. When wheat was grown in a rotation with clover and oats, no material loss of humus from the soil occurred. When the nitrogen and humus of the soil were conserved by the rotation of crops, and the production of clover, an increase of 20 bushels per acre of corn and 5.6 bushels of wheat were secured.

Lyon and Bizzell (6) have shown an average annual loss of nitrogen in the drainage waters from three soil tanks kept free from vegetation amounting to 92 pounds per acre. The average loss through drainage and the removal of crops, from seven cropped tanks (no clover), during the same period, was 70 pounds per acre.

These tanks, both the cropped and those kept bare, received during the 5 years, two applications of farm manure amounting to 10 tons per acre each, which furnished a total of approximately 200 pounds of nitrogen per acre for the 5 years, or 40 pounds per year. No estimate was made of the amount of nitrogen lost through volatilization of gaseous nitrogen or ammonia.

It would thus appear that the soil in these tanks is losing nitrogen at the rate of 30 to 52 pounds per acre annually, aside from any loss that may occur through the volatilization of gaseous nitrogen or ammonia.

By the analysis of samples of soil from large drainage tanks in which orange trees were growing, Collison and Walker (2) have shown an average loss of nitrogen from three fertilized tanks (tank no. 2 is omitted inasmuch as a legume crop was grown on this) amounting to 525 pounds per acre for a period of 5 years, or an average annual loss of 105 pounds per acre. This loss was arrived at by noting the difference between the nitrogen content of the original soil and its content at the end of the 5-year period. It therefore includes loss through drainage waters, any nitrogen that the growing orange tree may have utilized and any loss through the volatilization of gaseous nitrogen or ammonia. Unfortunately, the report does not state the amount of nitrogen that was applied during the 5 years nor the amount of nitrogen that was lost in the *drainage* waters.

The conditions which obtain in the majority of our cylinders, and in the lysimeter experiments referred to are just the conditions which Russell says favor the decomposition of organic matter and the disappearance of nitrogen: namely, copious aeration (which implies good drainage) and the presence of large quantities of organic matter.

#### LOSS OF NITROGEN FOR THE SECOND TEN-YEAR PERIOD

The loss of nitrogen for the third 5-year period (calculated separately for the three cylinders of each series) is shown in table 2. It is a striking coincidence that the average loss for the *A*'s and *B*'s for the 5 years is just half of



TABLE 2  
Loss of nitrogen during the third rotation

SERIES	a NITROGEN PRESENT, 1907	b NITROGEN APPLIED, 1908-1912	c m (a + b)	c NITROGEN REMOVED BY CROPS 1908-1912	d NITROGEN PRESENT, 1912	n (c + d)	(m-n) NITROGEN LOSS, EX- CLUSIVE OF CROPS 1908-1912
Section A							
	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	109.26	0.00	109.26	2.62	93.46	96.08	13.18
2	107.60	0.00	107.60	4.11	90.46	94.57	13.03
3	124.98	20.00	144.98	9.04	118.66	127.70	17.28
4	128.69	20.00	148.69	9.75	133.75	143.50	5.19
5	144.49	20.00	164.49	9.27	133.35	142.62	21.87
6	146.55	20.00	166.55	9.97	144.81	154.78	11.77
7	120.63	3.87	124.50	6.17	106.18	112.35	12.15
8	114.71	7.71	122.42	9.27	104.52	113.79	8.63
9	136.59	23.87	160.46	11.75	131.69	143.44	17.02
10	144.81	27.71	172.52	14.58	131.69	146.27	26.25
11	142.20	23.87	166.07	13.12	130.59	143.71	22.36
12	136.59	27.71	164.30	14.23	128.14	142.37	21.93
13	146.15	23.87	170.02	11.52	137.38	148.90	21.12
14	144.25	27.71	171.96	14.29	138.72	153.01	18.95
15	146.31	23.87	170.18	12.78	142.60	155.38	14.80
16	146.86	27.71	174.57	14.99	157.84	172.83	1.74
17	123.87	8.18	132.05	6.58	104.91	111.49	20.56
18	124.19	8.42	132.61	7.33	117.00	124.33	8.28
19	155.00	28.18	183.18	13.62	146.47	160.09	23.09
20	151.36	28.42	179.78	13.27	144.41	157.68	22.10
Average.....	134.75		153.31	10.41	126.83	137.24	16.06
Section B							
	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	107.60	0.00	107.60	4.28	91.80	96.02	11.58
2	109.81	0.00	109.81	5.72	89.27	94.99	14.82
3	139.20	20.00	159.20	11.23	127.66	138.89	20.31
4	133.98	20.00	153.98	11.30	121.58	132.88	21.10
5	135.88	20.00	155.88	10.38	136.75	147.13	8.75
6	144.81	20.00	164.81	11.34	137.07	148.41	16.40
7	113.76	3.87	117.63	7.97	104.52	112.49	5.14
8	114.08	7.71	121.79	11.07	103.49	114.56	7.23
9	133.83	23.87	157.70	13.57	129.24	142.81	14.89
10	135.88	27.71	163.59	16.43	133.91	150.34	13.25
11	135.88	23.87	159.75	16.35	125.06	141.41	18.34
12	131.40	27.71	159.11	16.49	126.95	143.44	15.67
13	145.60	23.87	169.47	15.02	132.40	147.42	22.05
14	155.71	27.71	183.42	15.30	139.99	155.29	28.13
15	146.15	23.87	170.02	13.87	134.85	148.72	21.30
16	144.49	27.71	172.20	16.41	137.62	154.03	18.17
17	124.27	8.18	132.45	9.28	111.47	120.75	11.70
18	118.26	8.42	126.68	8.74	112.26	121.00	5.68
19	158.47	28.18	186.65	15.24	149.07	164.31	22.34
20	153.66	28.42	182.08	15.60	139.75	155.35	26.73
Average.....	134.14		152.69	12.28	124.24	136.51	16.18



TABLE 2—Continued

## Section C

SERIES	a	b	m (a + b)	c	d	n (c + d)	(m - n) NITROGEN LOSS, EX- CLUSIVE OF CROPS 1908-1912	A's, B's, C's	
	NITROGEN PRESENT, 1907	NITROGEN APPLIED, 1908-1912		NITROGEN REMOVED BY CROPS, 1908-1912	NITROGEN PRESENT 1912			Average amount of nitrogen applied	Average amount of nitrogen lost
	gm.	gm.	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	109.26	0.00	109.26	5.59	92.90	98.49	10.77	0.00	11.84
2	116.13	0.00	116.13	7.24	102.30	109.54	6.59	0.00	11.48
3	149.15	20.00	169.15	12.09	136.28	148.37	20.78	20.00	19.46
4	133.83	20.00	153.83	12.92	140.38	153.30	0.53	20.00	8.94
5	146.86	20.00	166.86	14.59	150.10	164.69	2.17	20.00	10.93
6	142.44	20.00	162.44	14.07	138.49	152.56	9.88	20.00	12.68
7	116.13	3.87	120.00	9.56	106.49	116.05	3.95	3.87	7.08
8	120.87	7.71	128.58	12.43	109.57	122.00	6.58	7.71	7.48
9	145.44	23.87	169.31	17.22	142.12	159.34	9.97	23.87	13.96
10	144.81	27.71	172.52	18.87	142.12	160.99	11.53	27.71	17.01
11	137.14	23.87	161.01	16.91	134.46	151.37	9.64	23.87	16.78
12	145.44	27.71	173.15	20.41	140.54	160.95	12.20	27.71	16.60
13	158.47	23.87	182.34	16.76	145.20	161.96	20.38	23.87	21.18
14	149.55	27.71	177.26	18.08	145.99	164.07	13.19	27.71	20.09
15	150.81	23.87	174.68	17.15	146.55	163.70	10.98	23.87	15.69
16	152.47	27.71	180.18	17.82	146.94	164.76	15.42	27.71	11.78
17	125.69	8.18	133.87	10.84	118.66	129.50	4.37	8.18	12.21
18	127.74	8.42	136.16	10.26	115.89	126.15	10.01	8.42	7.99
19	156.42	28.18	184.60	17.18	156.02	173.20	11.40	28.18	18.94
20	153.66	28.42	182.08	18.51	143.23	161.74	20.34	28.42	23.06
Average...	139.12		157.67	14.43	132.71	147.14	10.534		14.26

the average loss for the first 10 years, that is, the annual rate of loss for the third 5 years is exactly the same as for the first 10 years, namely 103 pounds per acre.

The *apparent* average loss for the C's is less by a little more than one-third. The *apparent* loss is referred to here for the reason that these are the cylinders on which a legume is grown for green manure, and they therefore have at their disposal a certain amount of nitrogen in excess of the amount at the disposal of the A's and B's which amount cannot be definitely measured. For this reason, the figures, which are obtained by an indirect process, probably do not represent the full amount of the loss.

For this period those cylinders that receive the smaller applications of nitrogen generally show less loss than those that receive the heavy applications. The greatest loss, in most cases, is sustained by those cylinders that receive the manure or the manure and nitrate in combination.

The data for the fourth 5-year period are shown in table 3. Three out of the 20A cylinders made slight gains. There appears to be no particular reason why the three cylinders should have made gains when all the others

TABLE 3  
Loss of nitrogen during the fourth rotation

SERIES	a PRESENT 1913	b APPLIED 1913-1917	m (a + b) TOTAL PRESENT AND APPLIED	c REMOVED 1913-1917	d PRESENT 1917	n (c + d) TOTAL RE- MOVED AND PRESENT	m - n LOSS EX- CLUSIVE OF CROPS 1913-1917
Section A							
	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	93.46	0.00	93.46	1.75	87.30	89.05	4.41
2	90.46	0.00	90.46	2.39	83.32	84.71	5.75
3	118.66	20.06	138.72	8.95	119.69	128.64	10.08
4	133.75	20.06	153.81	9.72	120.08	129.80	24.01
5	133.35	20.06	153.41	8.51	131.46	139.97	13.44
6	144.81	20.06	164.87	9.10	140.23	149.33	15.54
7	106.18	3.85	110.03	4.16	94.01	98.17	11.86
8	104.52	7.70	112.22	5.07	101.04	106.11	6.11
9	131.69	23.91	155.60	9.95	150.42	160.37	4.77†
10	131.69	27.76	159.45	12.65	143.54	156.19	3.26
11	130.59	23.91	154.50	12.53	132.48	145.01	9.49
12	128.14	27.76	155.90	13.68	142.44	156.12	0.22†
13	137.38	23.91	161.29	10.25	144.73	154.98	6.31
14	138.72	27.76	166.48	13.23	153.81	167.04	0.56†
15	142.60	23.91	166.51	11.90	150.34	162.24	4.27
16	157.84	27.76	185.60	14.23	157.53	171.76	13.84
17	104.91	7.70	112.61	4.46	87.45	91.91	20.70
18	117.00	7.70	124.70	5.08	104.68	109.76	14.94
19	146.47	27.76	174.23	12.00	148.60	160.60	13.63
20	144.41	27.76	172.17	10.88	144.02	154.90	17.27
Average.....	126.83	20.52*	145.30	9.03	126.81	135.83	11.46†
Section B							
	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	91.80	0.00	91.80	4.39	82.32	86.71	5.09
2	89.27	0.00	89.27	4.77	83.90	88.67	0.60
3	127.66	20.06	147.72	10.14	124.19	134.33	13.39
4	121.58	20.06	141.64	11.86	120.00	131.86	9.78
5	136.75	20.06	156.81	10.38	128.77	139.15	17.66
6	137.07	20.06	157.13	11.01	142.28	153.29	3.84
7	104.52	3.85	108.37	7.40	92.67	100.07	8.30
8	103.49	7.70	111.19	9.33	94.09	103.42	7.77
9	129.24	23.91	153.15	11.78	134.40	146.18	6.97
10	133.91	27.76	161.67	14.12	138.41	152.53	9.14
11	125.06	23.91	148.97	15.26	124.19	139.45	9.52
12	126.95	27.76	154.71	14.96	129.17	144.13	10.58
13	132.40	23.91	156.31	12.87	133.98	146.85	9.46
14	139.99	27.76	167.75	13.23	141.57	154.80	12.95
15	134.85	23.91	158.76	12.77	142.60	155.37	3.39
16	137.62	27.76	165.38	14.30	136.99	151.29	14.09
17	111.47	7.70	119.17	8.39	104.91	113.30	5.87
18	112.26	7.70	119.96	7.77	104.12	111.89	8.07
19	149.07	27.76	176.83	12.57	140.94	153.51	23.32
20	139.75	27.76	167.51	13.78	140.07	153.85	13.66
Average.....	124.24	20.52*	142.71	11.05	121.98	133.03	9.67

\* Average of 18 determinations.

† Gain.

† Average of 17 determinations.

TABLE 3—Continued

SERIES	a	b	m (a + b) TOTAL PRESENT AND APPLIED	c	d	n (c + d) TOTAL RE- MOVED AND PRESENT	m - n EXCLUSIVE OF CROPS	
	PRESENT 1913	APPLIED 1913-1917		REMOVED 1913-1917	PRESENT 1917		Loss	Gain
Section C								
	gm.	gm.	gm.	gm.	gm.	gm.	gm.	gm.
1	92.90	0.00	92.90	6.67	90.69	97.36		4.46
2	102.30	0.00	102.30	8.96	97.96	106.92		4.62
3	136.28	20.06	156.34	13.41	143.07	156.48		0.14
4	140.38	20.06	160.44	15.03	134.70	149.73	10.71	
5	150.10	20.06	170.16	14.35	144.73	159.08	11.08	
6	138.49	20.06	158.55	15.61	150.50	166.11		7.56
7	106.49	3.85	110.34	10.97	107.91	118.88		8.54
8	109.57	7.70	117.27	12.74	99.62	112.36	4.91	
9	142.12	23.91	166.03	15.42	145.52	160.94	5.09	
10	142.12	27.76	169.88	16.51	142.44	158.95	10.93	
11	134.46	23.91	158.37	16.86	136.35	153.21	5.16	
12	140.54	27.76	168.30	20.16	141.25	161.41	6.89	
13	145.20	23.91	169.11	14.69	151.60	166.29	2.82	
14	145.99	27.76	173.75	17.10	162.50	179.60		5.85
15	146.55	23.91	170.46	16.19	157.45	173.64		3.18
16	146.94	27.76	174.70	17.40	151.13	168.53	6.17	
17	118.66	7.70	126.36	11.37	115.74	127.11		0.75
18	115.89	7.70	123.59	11.87	107.20	119.07	4.52	
19	156.02	27.76	183.78	16.86	156.97	173.83	9.95	
20	143.23	27.76	170.99	17.53	162.90	180.43		9.44
Average.....	132.71	20.52*	151.18	14.49	135.01	149.50	7.11†	

\* Average of 18 determinations.

† Average of 11 determinations.

were losing. The average loss for the remaining seventeen is 11.46 gm. equivalent to 73.3 pounds per acre, or approximately two-thirds of the amount lost during the third 5 years.

A loss is shown for all of the B's for the fourth period, though in the case of 2B it is almost negligible. It would appear that here, too, the nitrogen equilibrium, of which Russell speaks, has been reached. The heaviest loss—23.32 gm.—occurs with 19B which receives manure and ammonium sulfate. All cylinders from 9B to 16B, inclusive, receive like amounts of nitrogen in cow manure, and in addition the odd numbers receive 5 gm. of nitrate of soda and the even numbers 10 gm., and in this connection it may be pointed out that the latter show a greater loss than the former.

The average loss for the entire series is 9.67 gm. equivalent to a yearly average of 61.9 pounds per acre. Of the C's 11 show losses averaging 7.11 gm. and 9 show gains averaging 4.99 gm. In the case of 3C and 17C the gains are only slight and here again an equilibrium appears to have been reached.

As previously explained these gains are apparent rather than real for the reason that an unknown amount of atmospheric nitrogen has been supplied by the two legume crops, and this has not been included in the nitrogen applied.

#### PERCENTAGE OF NITROGEN IN SOILS

In connection with the foregoing discussion it is of interest to study the figures showing the percentage of nitrogen in the original soil used in these cylinders, in comparison with the composition of samples drawn from the various cylinders at 5-year intervals. These figures are shown in table 4. On account of the difference in treatment during the second 10-year period it is necessary to report the *A*, *B*, and *C* sections separately. It will be noted that in most cases these figures show a general decline in nitrogen content. In a few cases, notably 14*A*, 15*A* and 16*A*, the nitrogen content has been kept well up to the original condition. The same is true of three or four of the cylinders in section *C*, but on account of the legume crops which were grown in this section, they cannot fairly be considered in connection with the two other sections.

The rather rapid decline in nitrogen content of series 1 and 2 is not surprising. These cylinders, it will be remembered, produce crops each year without having any nitrogen added. In the 20 years then, the nitrogen content has dropped from nearly 0.2 per cent to about 0.10 to 0.11 per cent. No. 7, 8, 17 and 18 also have lost heavily. These are the cylinders that receive all their nitrogen in the form of commercial fertilizers and do not receive any farm manure, and with the exception of those in section *C*, do not receive any green manure.

This is a good illustration of what happens when land is continually under cultivation without the use of green-manure crops or farm manures to aid in maintaining the supply of organic matter. Even the green-manure crop cylinders show a rather heavy loss.

It may be pointed out that during the period of 1912 to 1918 the losses were not very great in most cases. Indeed in some cases there were fair increases in nitrogen content. Taking the averages for all the series it is found that the nitrogen content remained very nearly constant during this period. This must not be taken as a contradiction of results reported in table 3 where a loss of nitrogen is shown for this same period, for it must be remembered that nearly twice as much nitrogen was applied as was removed by the crops during the 5 years.

It is possible that the soil in series 1 and 2 has reached an equilibrium which may be maintained for a number of years, though this could not go on indefinitely if the land is continuously cropped without the use of nitrogenous fertilizers or green manures.

It is of interest to note that the soils of section *B* show a lower nitrogen content than those of either of the other sections.

TABLE 4  
Percentage of nitrogen present, air-dry soil—1898, 1907, 1912, 1918

SERIES	SECTION A				SECTION B				SECTION C			
	Nitrogen originally present	Present in 1907	Present in 1912	Present in 1918	Originally present	Present in 1907	Present in 1912	Present in 1918	Originally present	Present in 1907	Present in 1912	Present in 1918
	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent	per cent
1	0.1968	0.1383	0.1183	0.1105	0.1968	0.1362	0.1162	0.1042	0.1968	0.1383	0.1176	0.1148
2	0.1968	0.1362	0.1145	0.1042	0.1968	0.1390	0.1130	0.1062	0.1968	0.1470	0.1295	0.1240
3	0.1968	0.1582	0.1502	0.1515	0.1968	0.1762	0.1616	0.1572	0.1968	0.1888	0.1725	0.1811
4	0.1968	0.1629	0.1693	0.1520	0.1968	0.1696	0.1539	0.1519	0.1968	0.1694	0.1777	0.1705
5	0.1968	0.1829	0.1688	0.1664	0.1968	0.1720	0.1731	0.1630	0.1968	0.1859	0.1900	0.1832
6	0.1968	0.1855	0.1833	0.1775	0.1968	0.1833	0.1735	0.1801	0.1968	0.1803	0.1753	0.1905
7	0.1968	0.1527	0.1344	0.1190	0.1968	0.1440	0.1323	0.1173	0.1968	0.1470	0.1348	0.1366
8	0.1968	0.1452	0.1323	0.1279	0.1968	0.1444	0.1310	0.1191	0.1968	0.1530	0.1387	0.1261
9	0.1968	0.1729	0.1667	0.1904	0.1968	0.1694	0.1636	0.1701	0.1968	0.1841	0.1799	0.1842
10	0.1968	0.1833	0.1667	0.1817	0.1968	0.1720	0.1695	0.1752	0.1968	0.1833	0.1799	0.1803
11	0.1968	0.1800	0.1653	0.1677	0.1968	0.1720	0.1583	0.1572	0.1968	0.1736	0.1702	0.1726
12	0.1968	0.1729	0.1622	0.1803	0.1968	0.1660	0.1607	0.1635	0.1968	0.1841	0.1779	0.1788
13	0.1968	0.1850	0.1739	0.1832	0.1968	0.1843	0.1676	0.1696	0.1968	0.2006	0.1838	0.1919
14	0.1968	0.1826	0.1756	0.1947	0.1968	0.1971	0.1772	0.1792	0.1968	0.1893	0.1848	0.2057
15	0.1968	0.1852	0.1805	0.1903	0.1968	0.1850	0.1707	0.1805	0.1968	0.1909	0.1855	0.1993
16	0.1968	0.1859	0.1998	0.1994	0.1968	0.1829	0.1742	0.1734	0.1968	0.1930	0.1860	0.1913
17	0.1968	0.1568	0.1328	0.1107	0.1968	0.1573	0.1411	0.1328	0.1968	0.1591	0.1502	0.1465
18	0.1968	0.1572	0.1481	0.1325	0.1968	0.1497	0.1421	0.1318	0.1968	0.1617	0.1467	0.1357
19	0.1968	0.1962	0.1854	0.1881	0.1968	0.2006	0.1887	0.1784	0.1968	0.1980	0.1975	0.1987
20	0.1968	0.1916	0.1828	0.1823	0.1968	0.1945	0.1769	0.1773	0.1968	0.1945	0.1813	0.2062
Average.....	0.1968	0.1706	0.1605	0.1605	0.1968	0.1698	0.1573	0.1544	0.1968	0.1761	0.1680	0.1709

This should probably be attributed to the more rapid oxidation of the organic matter in the presence of lime. Lime was applied also to section C but the legume green-manure crops that are grown on this section have tended to raise the nitrogen content above that of the other two sections.

#### THE CARBON CONTENT OF CYLINDER SOILS

Carbon determinations were made on the soils collected in 1918, and are reported in table 5. A comparison of the carbon content at this time, with

TABLE 5  
*Percentage of carbon in cylinder soils—1918*

SERIES	SECTION A	SECTION B	SECTION C
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
1	1.390	1.360	1.630
2	1.350	1.285	1.670
3	2.005	1.975	2.365
4	1.835	1.730	2.105
5	1.955	2.010	2.285
6	2.045	2.135	2.500
7	1.235	1.335	1.745
8	1.305	1.375	1.740
9	2.245	2.175	2.300
10	2.260	2.225	2.450
11	1.975	1.895	2.295
12	2.140	1.790	2.340
13	2.210	2.135	2.460
14	2.445	2.135	2.555
15	2.320	2.175	2.405
16	2.340	2.095	2.310
17	1.280	1.595	1.725
18	1.405	1.540	1.795
19	2.300	2.220	2.455
20	2.090	2.085	2.360
Average.....	1.907	1.864	2.175

the carbon content of the original soil, is instructive. A determination made on a sample of the original soil showed 1.83 per cent of carbon.

It will be noted that in a number of cases the carbon content has increased, while in a few cases it has decreased. Those cylinders that have received manure alone have increased but slightly in carbon content; those that have received both manure and nitrate of soda, in most cases have made considerable gains. In section A those that receive the 10-gm. portion of nitrate—no. 10, 12, 14 and 16—have made greater gains than those that receive the 5-gm. portion—no. 9, 11, 13 and 15.

Those in sections *A* and *B* that receive no nitrogen and also those that receive commercial nitrogenous materials only, have lost notably. On section *C* the green-manure crop has aided in keeping up the carbon supply.

Attention may be called to the fact that the average carbon content of the soils in section *B* is less than the average in section *A*.

This would seem to be further evidence that the lime favored the oxidation and disappearance of the organic matter, while the higher average on section *C* shows the value of a green-manure crop in maintaining the supply of organic matter.

There need not necessarily be any inconsistency in an increase in the carbon content of a soil, the nitrogen content of which is decreasing. With the decomposition of the less resistant part of the organic matter which contains much of the nitrogen, there may be an accumulation in the soil of the harder more resistant parts of the plant residues, thus increasing the carbon content.

#### SUMMARY

A study has been made of nitrogen losses from a loam soil (in cylinders with natural drainage) which has been under a 5-year rotation of corn, oats (2 years), wheat and timothy, for 20 years.

There are 60 of these cylinders arranged in 20 series of 3 cylinders each, and the fertilizer and manure treatment is the same for the 3 cylinders of a given series.

Different forms of nitrogenous fertilizers are used alone and also in combination with farm manure. All cylinders with the exception of those of series 1, receive liberal applications of phosphoric acid and potash annually.

During the first 10 years, the lime treatment was the same for all cylinders. Beginning with the second 10-year period, lime (ground limestone) has been applied to the second and third cylinders of each series at 5-year intervals. A further differentiation has been brought about by growing two legume green-manure crops on the third cylinder of each series in the course of each 5-year rotation. Thus the first cylinder of each series, or 20 cylinders designated as section *A*, have been without lime for 20 years.

A complete record has been kept of the amount of nitrogen applied in the form of fertilizers or manures, and also of the amount removed by the crops during this period. Also, nitrogen determinations have been made on samples of the original soil and on samples collected in 1907, 1912 and 1918. From these latter records, the loss of nitrogen from the soil, exclusive of that removed by the crops, has been calculated.

During the first 10 years, this amounted to 32.24 gm. per cylinder, or the equivalent of 103 pounds per acre annually. During the third 5-year period, the loss was one-half this amount (except in the legume section), or again 103 pounds per acre annually. During the fourth 5-year period, the average loss was considerably less, and for certain of the cylinders on sections *A* and *C* there was some gain. This gain on section *C* is due largely to the legume



crops that were grown on this section. There appears to be no particular reason for the gain on three of the cylinders of section A. This statement of gain, however, is not a contradiction of the statement that in most cases these soils have been constantly losing in nitrogen content, since with only one or two exceptions more nitrogen has been applied than has been removed by the crops.

Carbon determinations made on the set of samples collected in 1918 show gains in carbon content over the original soil, for nearly all those cylinders that receive both farm manure and commercial nitrogenous fertilizers. Those soils that receive farm manure alone have not been changed much in carbon content, while those that have received nitrogen in the form of commercial fertilizers only, or have received no nitrogen at all have lost appreciably in carbon content.

A gain in carbon is not necessarily inconsistent with a loss in nitrogen.

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#### PLATE 1

FIG. 1. WHEAT ON NITRATE OF SODA CYLINDERS (SERIES 8), 1916  
Compare A (no lime) with B and C (limed)

FIG. 2. WHEAT ON SULFATE OF AMMONIA CYLINDERS (SERIES 17), 1916

The continued use of sulfate of ammonia without lime has rendered soil practically sterile. Compare A with B and C.

FIG. 3. WHEAT ON MANURE AND SULFATE OF AMMONIA CYLINDERS (SERIES 19), 1916  
Manure appears to aid in overcoming the unfavorable condition produced by sulfate of ammonia when used alone; compare A with A in figure 2.

Note: A's unlimed; B's and C's limed at intervals of 5 years; C's produce two legume green-manure crops every 5 years.

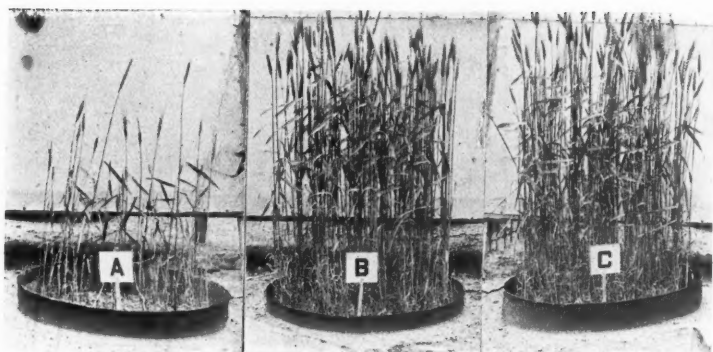


FIG. 1

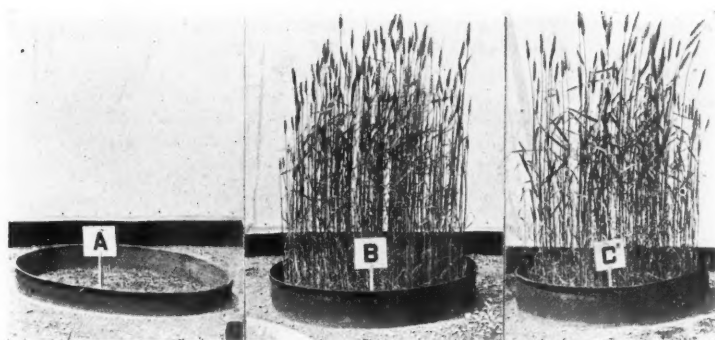


FIG. 2

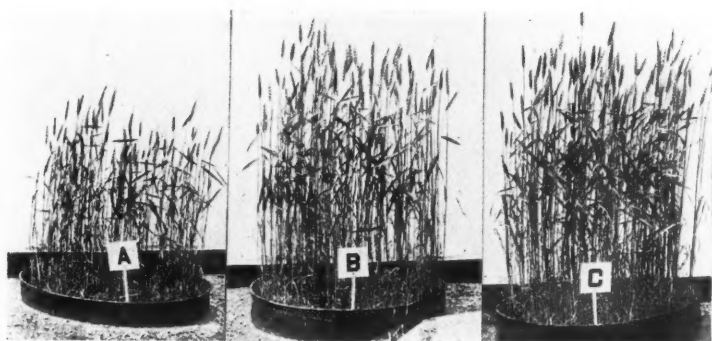


FIG. 3

## PLATE 2

### FIG. 1. TIMOTHY ON "NO FERTILIZER" CYLINDERS (SERIES 1), 1917

20 years of cropping without fertilizer or lime has left the soil without producing power; compare *A* with *B* and *C*.

### FIG. 2. TIMOTHY ON CHECK CYLINDERS (SERIES 2), 1917

Minerals annually, but no nitrogen applied; nitrogen long ago became the limiting factor (see *A*), but when lime is applied a moderate crop is still produced (see *B* and *C*).

### FIG. 3. TIMOTHY ON MANURE CYLINDERS (SERIES 3), 1917

Manure supplies the needed nitrogen and appears also to satisfy the need for lime; compare *A* with *B* and *C*.

*Note:* *A*'s unlimed; *B*'s and *C*'s limed at intervals of 5 years; *C*'s produce two legume green-manure crops every 5 years.

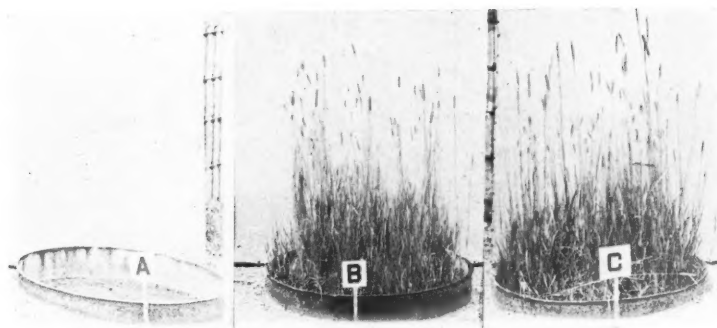


FIG. 1

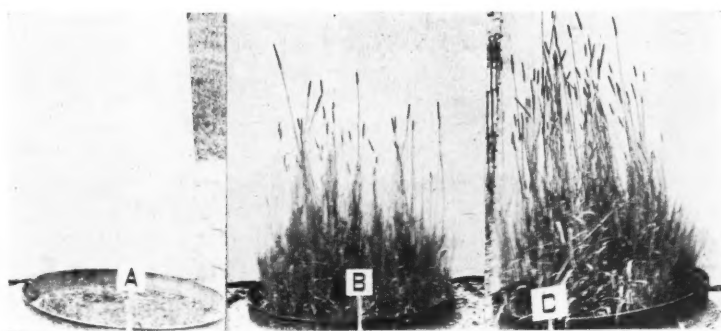


FIG. 2

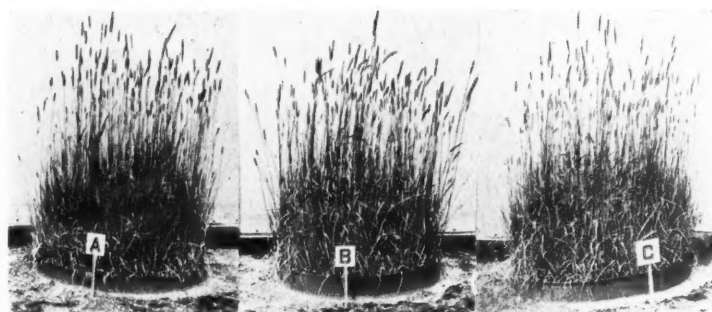
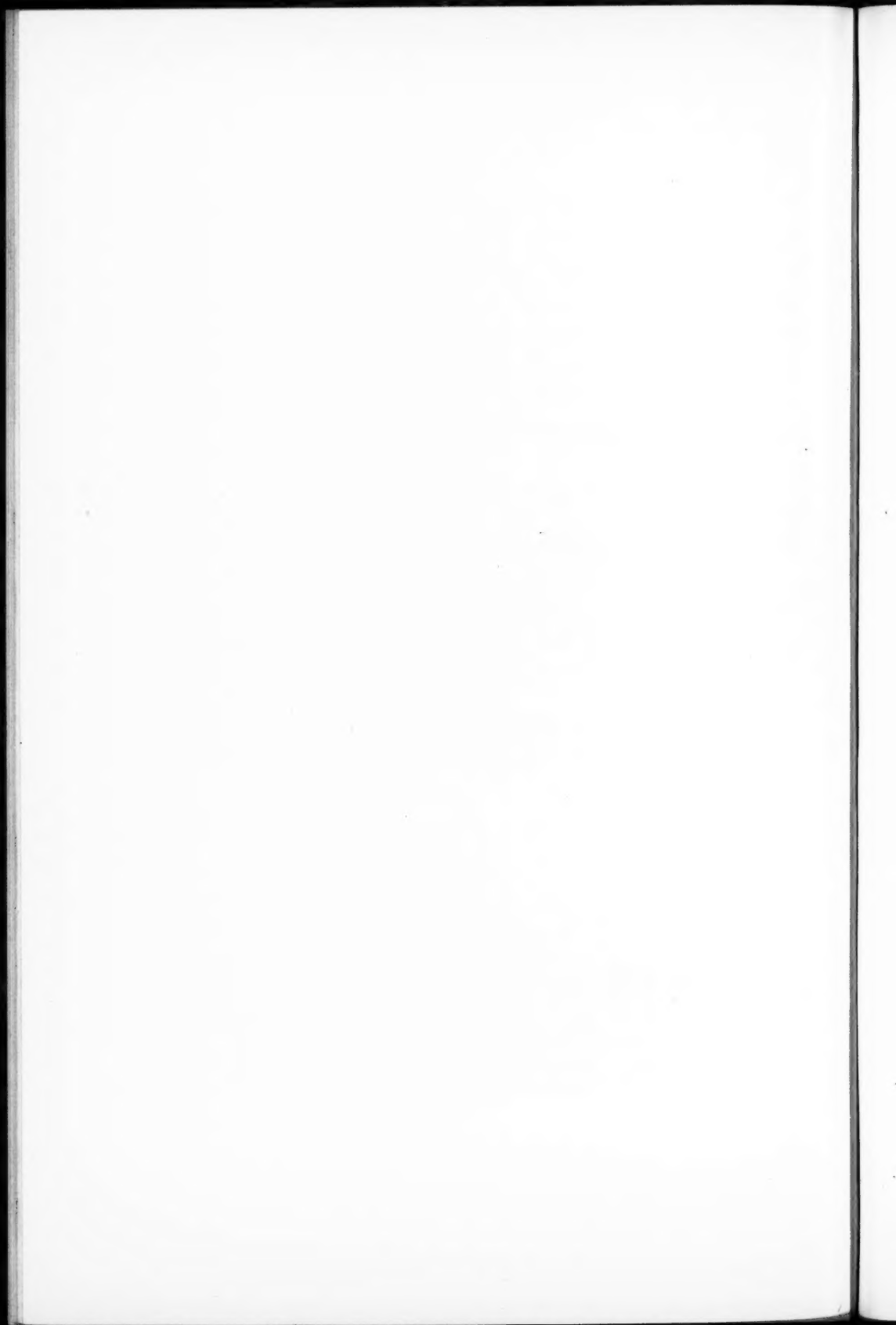


FIG. 3



# THE RELATION OF ORGANIC MATTER AND THE FEEDING POWER OF PLANTS TO THE UTILIZATION OF ROCK PHOSPHATE<sup>1</sup>

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The processes accompanying the decomposition of organic matter and the feeding power of plants are undoubtedly important factors in the utilization of rock phosphate. Those who strongly advocate the use of rock phosphate for soil improvement always advise that it be applied in intimate contact with decaying organic matter (13). The explanation usually given for this practice is that the organic acids, carbon dioxide and nitrous acid, resulting from such decomposition, are active agents in making the phosphate available. Strangely, however, when attempts have been made in the laboratory (12, 22, 31, 42) to prove this explanation, positive results have usually not been obtained, although in field tests rock phosphate has usually given the best results when used in connection with considerable organic matter. On the other hand, many investigators (5, 15, 17, 21, 24, 25, 28, 32, 33, 34, 37, 38) have observed that the growing plant itself possesses more or less power to feed directly on rock phosphate and that some plants possess especially marked powers. As a rule it has been found that most of the cereals are among the poor feeders, while the legumes, cruciferae and buckwheat are among the good feeders. The possibility of planning systems of farming in which the strong feeders may be used advantageously for an effective utilization of rock phosphate is of much practical importance.

## HISTORICAL REVIEW

In explanation of the difficulty of showing that decaying organic matter does make rock phosphate available Truog (42) suggested that the particles of rock phosphate and also the particles of the finely divided reprecipitated phosphate may be protected by slimy films of organic matter from extraction by the methods employed in the laboratory. He stated further that laboratory

<sup>1</sup> Part II of thesis submitted at the University of Wisconsin in partial fulfillment of the requirements for the Degree of Doctor of Philosophy. Published with the permission of the Director of the Wisconsin Agricultural Experiment Station.

The writer wishes to express his appreciation for the helpful suggestions and criticisms tendered by Professor E. Truog.

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composting experiments do not imitate field conditions and thus the efficiency of the carbon dioxide as a solvent may be lowered. He concluded that the decay of organic matter does increase the availability of rock phosphate by favoring a more thorough mechanical distribution of the phosphate in the soil, and the biological processes that give rise to the formation of carbon dioxide.

Tottingham and Hoffman (41) found that the amount of water-soluble phosphorus in fermenting mixtures of manure and rock phosphate decreased over periods of 4 to 6 months. They attributed this decrease, in part at least, to the use of soluble phosphorus by the bacteria themselves. They concluded that the final results from the mixing of manure and rock phosphate are advantageous.

Hopkins and Whiting (14) found that nitrite bacteria have the power of oxidizing ammonium sulfate in solution cultures containing pure tri-calcium phosphate, but no carbonate or free base, and that the nitrous and sulfuric acids resulting bring phosphorus into solution. The process is not accompanied with changes in acidity. They suggested the possibility of similar effects in the soil resulting from the decomposition of nitrogenous organic matter.

Truog (42) found that fermenting mixtures of manure and June grass became more alkaline as decomposition proceeded and pointed out that acids formed from decaying organic matter are likely to be neutralized before they could affect the rock phosphate.

Kelley (16) found that the calcium of rock phosphate does not furnish the calcium for nitrification under soil conditions and concluded that the decomposition of organic matter from the standpoint of nitrification has little effect on the availability of rock phosphate.

The earlier views held that plants excrete acids from their roots, which in themselves are the effective agents in the solution of mineral nutrients, like phosphates. In 1902 Kossowitsch (18) performed his famous experiment showing that the plant roots must be in contact with the rock phosphate in order to secure the phosphorus. Dyer (10), Quartaroli (36), Pfeiffer and Blanck (30), Palladin (27) and Maze (23) expressed their belief in the acid excretion theory. Shulov (39) found that ammonium nitrate favored the excretion of malic acid from plant roots, and thus brought rock phosphate into solution. Carso (6), though not stating definitely that acids are excreted from plant roots, did claim that the feeding power of plants of the same family for rock phosphate varies to some extent according to the acid content of the plant. Bogdanov (4), Prianishnikov (35), Chirikov (8), and Haas (11), however, claimed that no acids other than carbonic are excreted, and therefore no solvent power except that due to the carbon dioxide exists.

Kossowitsch (19, 20) and Stoklasa and Ernst (40) studied the carbon-dioxide excretion of roots of certain plants and found it to be quite large. Kossowitsch detected no relation between the feeding power of plants and the amount of carbon dioxide excreted. None of these experiments, however,



were carried out under natural and uniform conditions, and indicate a field for further research. Bogdanov (4) claimed that since the amount of carbon dioxide normally present in the soil is large the carbon dioxide excreted by plants would have but little additional solvent effect on rock phosphate. Truog (42), however, believed that the carbon dioxide excreted by the plant roots works at maximum efficiency in dissolving rock phosphate because of the removal of the soluble products. When carbon dioxide was used in culture experiments (26, 29, 45) only negative results on the solution of insoluble phosphates were secured.

In 1914 and 1916 Chirikov (7, 8) published the results of his experiments from which he concluded that plants which more energetically assimilate calcium are the better feeders on rock phosphate. He stated that when the ratio of CaO to  $P_2O_5$  in the plant is greater than 3 the plant is usually a good feeder. In 1915 and 1916 Truog (43, 44) put forth the theory that it is the CaO content of the plant and not the ratio of CaO to  $P_2O_5$  that bears an important relation to the feeding powers of plants.

#### EXPERIMENTAL

##### *Materials used*

The soil medium consisted of either sand or soil. The sand was practically pure white quartz and contained no soluble phosphorus. The soil was a strongly acid Plainfield sand, which in the air-dry condition contained 0.59 parts per million of water-soluble phosphorus.

The different forms of organic matter used consisted of finely pulverized air-dry buckwheat hay, sweet clover hay, alfalfa hay, soybean hay, mammoth clover hay, corn stover, oats straw, and chopped green buckwheat and sweet clover.

The rock phosphate used was a finely ground brown rock containing 15.4 per cent of total phosphorus, 0.025 per cent of water-soluble phosphorus and 0.076 per cent of phosphorus soluble in water saturated with carbon dioxide. The acid phosphate was a commercial product containing 6.5 per cent of phosphorus. The limestone was a pulverized dolomite containing a large amount of fine material. Unless otherwise specified these materials were used at the rate of 1 ton, 400 pounds and 2 tons to the acre, respectively.

The nutrient solution used in the culture experiments was the same as that employed in the experiments previously reported (3) in which  $NaNO_3$  was used as the nitrogen salt. The pot cultures described herein were managed in the same manner as those in the experiments just referred to.

##### *The effect of organic matter without leaching on the availability of rock phosphate*

Manure has usually been employed as the form of organic matter in studies of this kind. Blood meal, June grass and cottonseed meal have also been used. No record was found of studies with other residues which a farmer

might use in this connection. Consequently some preliminary studies were made with sweet clover, mammoth clover and buckwheat hay.

The work was carried on in Mason fruit jars containing 500 gm. of either sand or soil. Applications in accordance with the arrangement indicated in table 1 were made as follows on the acre basis: organic matter 5 tons, rock phosphate 1 ton and limestone 2 tons. The soluble phosphorus was determined in both the organic matter and phosphate used. At the time of starting, 10 cc. of a filtered soil infusion was added to each jar. The moisture content was maintained at 13 per cent of the weight of the mixture. The mixtures were agitated frequently by vigorous shaking of the jars and allowed to remain in a dark place in the greenhouse. All mixtures were set up in duplicate.

TABLE 1

*The amounts of water-soluble phosphorus in each jar before and after decomposition of organic matter, in mixtures indicated*

FORMS OF ORGANIC MATTER	TREATMENTS	SAND MIXTURES			SOIL MIXTURES		
		Amount of soluble phosphorus		Decrease of soluble phosphorus	Amount of soluble phosphorus		Decrease of soluble phosphorus
		Beginning	End		Beginning	End	
		mgm.	mgm.	mgm.	mgm.	mgm.	mgm.
Sweet clover.....	None	5.45	1.90	3.55	5.74	1.90	3.84
	Rock phosphate	5.87	2.30	3.57	6.16	2.60	3.56
	Rock phosphate, lime	5.87	3.10	2.77	6.16	1.00	5.16
Mammoth clover...	None	5.40	2.70	2.70	5.69	1.30	4.39
	Rock phosphate	5.82	3.00	2.82	6.11	1.20	4.91
	Rock phosphate, lime	5.82	2.60	3.22	6.11	1.00	5.11
Buckwheat.....	None	6.55	0.40	6.15	6.84	0.70	6.14
	Rock phosphate	6.97	3.75	3.22	7.26	0.75	6.51
	Rock phosphate, lime	6.97	2.20	4.77	7.26	0.80	6.46

At the end of nearly 5 months the mixtures were brought to an air-dry condition, by drying rapidly in an oven and then exposing to laboratory conditions before analysis. Samples of 200 gm. were shaken vigorously by hand with 500 cc. of distilled water, allowed to settle and the supernatant liquid filtered off through Pasteur-Chamberland clay filters. The filtered solutions, after oxidation with bromine water to destroy the organic matter, were analyzed for phosphorus by the Pemberton method.

The results are recorded in table 1. The amounts of soluble phosphorus found were not large in any case and did not show any regularity to the treatments given. There was usually a slight increase of soluble phosphorus where rock phosphate was used in addition to the organic matter. In some cases the limestone appeared to increase slightly the solubility of the phosphate,

while in others, it caused a slight decrease. In accord with the results of previous investigators, in no case was there found to be as much soluble phosphorus at the end of the decomposition period as there was known to exist at the beginning. With all combinations the soluble phosphorus became more or less insoluble.

*The effect of organic matter with leaching on the availability of rock phosphate*

In view of the failure of experiments to show that the decomposition of organic matter makes rock phosphate available, it was thought that perhaps leaching the mixtures once a week would better imitate the conditions of the field and thus show a solvent effect. Two series of percolators containing sweet clover hay as the organic matter and other materials similar in kind

TABLE 2

*Water-soluble phosphorus, calcium and nitric nitrogen in leachings from series leached at intervals and series leached only at the end*

MEDIUM	TREATMENTS	SERIES LEACHED AT INTERVALS			SERIES LEACHED ONLY AT THE END		
		P	Ca	Nitric N	P	Ca	Nitric N
		p. p. m.	p. p. m.	p. p. m.	p. p. m.	p. p. m.	p. p. m.
Sand...	Phosphate	1.70	26.06	10.00	1.64	15.04	3.00
	Sweet clover	2.80	49.88	29.62	2.24	17.64	24.50
	Sweet clover, phosphate	2.30	46.90	18.62	1.82	16.84	26.50
	Sweet clover, phosphate, lime	2.32	45.90	20.60	1.70	17.84	24.50
Soil....	None	2.16	28.46	23.12	1.32	16.84	14.00
	Phosphate	2.20	28.46	23.60	1.32	18.85	15.50
	Sweet clover	2.26	25.84	31.10	0.98	35.89	49.00
	Sweet clover, phosphate	2.34	25.66	29.18	1.52	36.29	47.50
	Sweet clover, phosphate, lime	1.76	31.16	39.28	1.28	42.71	62.00

and quantity to those in the experiments just reported were arranged. One series was leached weekly with 200 cc. of distilled water for a period of 12 weeks, and the other only at the end. Each treatment was duplicated. The inoculated mixtures were kept in a dark place in the greenhouse and stirred and watered as needed. The leachings were filtered immediately through Pasteur-Chamberland clay filters. After a period of more than 3 months, the other series was leached with 200-cc. portions of distilled water until a total of 1200 cc. of leachings were secured. These were filtered in the same manner as the others. The filtered solutions from both series were analyzed for phosphorus, calcium, and nitric nitrogen. The phosphorus and calcium were determined volumetrically by the Pemberton and permanganate methods, respectively, after the organic matter in solution had been decomposed by bromine water. The nitric nitrogen was determined by Devarda's reduction method.

The results are given in table 2. The data of the two series are not strictly comparable on account of the fact that the total leachings from the series leached at intervals was twice as large as from the other. The amount of soluble phosphorus was not large in either series. There was but little difference between the influence of the quartz sand and of the Plainfield sand. The soluble calcium in the regularly leached series was higher in the sand than in the soil, but in the other series this was not true. Nitrification was greater in the soil than in the sand. The results failed to indicate a solvent action of the decaying organic matter on rock phosphate under either leached or unleached conditions. Neither can it be concluded from these results that rock phosphate becomes more available with greater nitrification.

*The effect of decaying organic matter on the availability of rock phosphate under the influence of moving capillary water*

Since it was impossible to prove a solvent action of decaying organic matter on rock phosphate under leached and unleached conditions, it was thought that perhaps different results might be obtained under conditions in which the soluble phosphate would be removed as formed from the field of action by capillary water. To test out this idea six galvanized cylinders 10 inches in diameter and 2 feet high were arranged so that the water could be supplied from the bottom. A layer of quartz sand was placed on the bottom of each cylinder to a depth of 3 inches. On top of this layer was placed a layer consisting of a mixture of sand, rock phosphate and green manure. Green buckwheat and green sweet clover were used as green manure. Limestone also was added to some of the mixtures. To each mixture was added 20 cc. of filtered soil infusion. The cylinders were then filled to within 3 inches of the top with a layer of very fine pure quartz sand. Bottles were attached to maintain automatically a water level inside the cylinders below the phosphate mixtures and thus make possible a continuous capillary stream from the bottom to the top of the cylinders where the water evaporated. Small holes were provided around the cylinders at the same level as the fermenting mixtures in order to provide aeration at that point. The cylinders were kept in the greenhouse in this condition for a period of almost 8 months. At the end of this time there still remained a dry layer of about 2 inches at the surface due to evaporation of the water before it came entirely to the surface. All the dry sand was carefully removed from above the moist layer. The moist layer, to a depth of 6 inches, was then removed, thoroughly mixed and dried, and analyzed for phosphorus and calcium as follows:

Samples of 600 gm. of the dried sand were placed in flasks and boiled with 250 cc. of 1:1  $\text{HNO}_3$ . The contents were then brought up to a volume of 750 cc. and allowed to stand until the supernatant liquid was perfectly clear. Duplicate aliquots of 250 cc. of the clear solution were taken for the analysis. The results are given in table 3.

These results show that soluble phosphorus and calcium were moved upward by the capillary water. In general, however, the presence of organic matter decreased the amount of soluble phosphorus and calcium moved upward. The slightly increased result with the combination of sweet clover and rock phosphate is within the limits of experimental error, and from the results obtained it cannot be said that the decay of organic matter has brought rock phosphate into solution.

TABLE 3

*The amounts of phosphorus and calcium moved upward by capillary water from mixtures decaying organic matter and rock phosphate in fine quartz sand*

TREATMENT	COMPOSITION OF DRY SOIL		POUNDS TO AN ACRE	
	P	Ca	P	Ca
	p. p. m.	p. p. m.	lbs.	lbs.
Rock phosphate.....	3.20	41.10	6.4	82.3
Buckwheat.....	3.15	40.60	6.3	81.2
Buckwheat, rock phosphate.....	3.00	32.08	6.0	64.7
Sweet clover.....	3.05	33.08	6.1	66.7
Sweet clover, rock phosphate.....	3.25	28.57	6.5	57.1
Sweet clover, rock phosphate, lime.....	2.95	21.05	5.9	42.1

The organic matter was used at the rate of 3 tons, rock phosphate 1 ton and limestone 2 tons to the acre.

*The effect of organic matter extracts on the solubility of rock phosphate*

In view of the fact that no direct solvent action could be demonstrated by composting rock phosphate in direct contact with organic matter, there remained the possibility of showing an action with the extracts of decaying organic matter. To provide the means for testing this possibility, 10 gm. each of pulverized air-dry sweet clover hay, mammoth clover hay, soybean hay, buckwheat hay, corn stover and oats straw were thoroughly mixed with 1000 gm. of pure quartz sand and placed in separate percolators. Each mixture was inoculated with 10 cc. of a filtered soil infusion and then maintained at a moisture content of 13 per cent. The mixtures were shaken vigorously at intervals and kept in a dark place in the greenhouse for a period of 6 weeks. At the end of this period each percolator was leached with 200-cc. portions of distilled water until a total of 2000 cc. of extract was secured. These extracts were filtered immediately through Pasteur-Chamberland clay filters and samples analyzed for phosphorus and calcium.

To test the solvent action of these extracts and also of distilled water on rock phosphate, a battery of percolators was arranged in two sets; so that tests could be made with and without the addition of carbon dioxide. The carbon dioxide was used in one set to make the conditions more comparable to that of the field. To each percolator was added 600 cc. of the extract and 4.6 gm. of rock phosphate, which is the proportion of rock phosphate and water

used in the decomposition experiments and in pot cultures carried on in this investigation. Effective agitation was maintained in the one case by the bubbling of  $\text{CO}_2$  from a tank, and in the other by the aspiration of  $\text{CO}_2$ -free air. After 48 hours of such contact at room temperature the extracts were siphoned off and filtered through Pasteur-Chamberland clay filters. The extracts were analyzed for phosphorus and calcium as before. Where the  $\text{CO}_2$  was used there was an appreciable disappearance of color in all solutions, indicating the precipitation of the soluble and colloidal organic matter in the solutions. The results of these studies are given in table 4.

Of the original extracts, the buckwheat extract contained the largest amount of soluble phosphorus and calcium, while the corn stover and oats-straw extracts contained the smallest amounts. In the absence of carbon dioxide, agitation of the extracts with rock phosphate decreased the amounts of soluble phosphorus and calcium, save in two cases where slight increases of soluble

TABLE 4

*The amounts of phosphorus and calcium in organic matter extracts before and after contact with rock phosphate and with and without carbon dioxide*

KIND OF EXTRACT OR LIQUID	ORIGINAL CONTENT		CONTENT AFTER CONTACT WITH ROCK PHOSPHATE			
			Without $\text{CO}_2$		With $\text{CO}_2$	
	P	Ca	P	Ca	P	Ca
	p. p. m.	p. p. m.	p. p. m.	p. p. m.	p. p. m.	p. p. m.
Distilled water.....			1.92	9.62	5.84	28.07
Buckwheat hay extract.....	7.10	39.09	4.80	14.44	5.20	26.47
Soybean hay extract.....	4.30	31.08	2.72	14.44	4.72	26.66
Sweet clover hay extract.....	4.05	22.05	3.12	13.23	4.84	24.06
Mammoth clover hay extract....	3.45	22.56	3.76	11.63	4.72	19.25
Corn stover extract.....	3.44	16.04	3.48	13.23	4.96	21.85
Oats straw extract.....	3.00	17.24	2.88	16.04	4.24	24.06

phosphorus occurred which were within the limits of experimental error. In the presence of carbon dioxide agitation of the extracts with rock phosphate gave no regular or definite increases of soluble calcium, but the soluble phosphorus increased in all cases except with the buckwheat extract. The distilled water alone dissolved an appreciable amount of phosphorus and calcium, and with carbon dioxide it dissolved more phosphorus and calcium than were found in solution in the extracts treated similarly. The presence of calcium carbonate as an impurity in the commercial rock phosphate may be a factor which causes the soluble phosphorus to go out of solution in fermenting mixtures of organic matter and rock phosphate. Undoubtedly, carbon dioxide changes some of this into calcium bicarbonate which may react with soluble phosphates and precipitate them. The contact of soluble tricalcium phosphate with soluble mono-calcium phosphate tends toward an equilibrium in which some of the soluble phosphate reverts to the more insoluble di-calcium



phosphate. DeTurk (8) in a somewhat similar manner found that the amount of soluble potash decreased in fermenting organic mixtures containing insoluble potash minerals. He explained his results as due to the direct absorption of the soluble potash by the minerals.

In considering the reasons for the decrease of the soluble phosphorus in fermenting mixtures, the use of soluble phosphorus by the bacteria, as pointed out by Tottingham and Hoffman (41) must not be overlooked. In the experiments with the organic liquids, however, it is not likely that bacteria had much influence. No changes due to nitrification could be detected in these extracts.

*The relation of decaying organic matter and rock phosphate to plant growth*

In view of the fact that chemical studies made with decaying organic matter did not show any solvent action on rock phosphate, the question still remained as to the effect of such mixtures on the growth of plants. This problem was investigated with a series of sand cultures in which corn was grown with several forms of organic matter in various combinations with rock phosphate and limestone, as indicated in table 5.

As sources of organic matter, finely pulverized alfalfa hay, buckwheat hay, oats straw and chopped green buckwheat hay were used. The dry materials contained 0.212, 0.282 and 0.113 per cent of phosphorus, respectively. The green buckwheat was not analyzed. Each kind of organic matter was used in two amounts. The larger application of each contained approximately 50 mgm. of phosphorus which was sufficient for a good growth of corn provided it could all be used. The smaller application was approximately one-third of the larger, which was inadequate for good growth, yet furnished sufficient phosphorus to give the plant a good start and enough organic matter to have a solvent effect on the rock phosphate. Checks were maintained without organic matter and without phosphorus. The nutrient solution used was the same as that employed in the sand cultures previously reported (3) in which  $\text{NaNO}_3$  was the form of nitrogen salt added, except that half of the  $\text{NaNO}_3$  was replaced with an equivalent amount of  $\text{Ca}(\text{NO}_3)_2$ . The cultures were managed in a manner similar to the method employed with previous ones with respect to moisture and other conditions. All cultures were duplicated.

Shortly after germination the corn showed evidences of toxic effects where the organic matter, with the exception of the green buckwheat, had been used. With the alfalfa and the dry buckwheat these effects disappeared after several weeks. Where the limestone was used the toxic effects were much less. The cultures with oats straw, however, never recovered and most of the plants finally perished. Because of lack of time, the plants were harvested after 60 days of growth. A longer period of growth would undoubtedly have shown more striking results. The yields of dry matter are given in table 5.



The results show that the phosphorus in the organic matter was readily available to growing corn. The dry buckwheat was by far the most efficient. When the application of buckwheat hay was reduced to one-third, the growth was also reduced to about one-third. The difference in the case of the alfalfa hay due to the amount used was not so marked. The largest growth of all occurred where the large quantity of dry buckwheat was applied.

All combinations of organic matter and rock phosphate gave substantial increases in the yield of dry matter over that produced by either the organic matter or rock phosphate when used alone. Increases from the combination

TABLE 5

*The amounts of dry matter produced in quartz sand corn cultures with mixtures of organic matter, rock phosphate and limestone*

FORM OF ORGANIC MATTER	AMOUNT OF ORGANIC MATTER USED	SUPPLEMENTARY TREATMENTS*	DRY MATTER PRODUCED			INCREASE OF TOTAL DRY MATTER OVER CHECK	INCREASE OF TOTAL DRY MATTER WITH MIXTURE OF ROCK PHOSPHATE AND ORGANIC MATTER OVER COMBINED SEPARATE INCREASES DUE TO THESE
			Tops	Roots	Total		
	gm.		gm.	gm.	gm.	gm.	gm.
None (check).	0	None	2.6	1.6	4.2		
	0	Rock phosphate	5.5	3.8	9.3	5.1	
Dry alfalfa hay.....	25	None	4.1	3.1	7.2	3.0	
	8	None	2.4	1.8	4.2	0.0	
	8	Rock phosphate	6.8	3.6	10.4	6.2	1.1
	8	Rock phosphate, lime	9.4	4.2	13.6	9.4	4.3
Dry buck-wheat hay..	20	None	16.0	5.1	21.1	16.9	
	6	None	4.6	2.2	6.8	2.6	
	6	Rock phosphate	11.2	5.5	16.7	12.5	4.8
	6	Rock phosphate, lime	7.6	4.3	11.9	7.7	0.0
Green buck-wheat tissues	50	None	5.8	4.2	10.0	5.8	
	50	Rock phosphate	8.6	6.2	14.8	10.6	-0.3

\* Rock phosphate used at the rate of 1 ton and limestone at the rate of 2 tons to the acre.

of organic matter and phosphate over the combined increases from these materials used separately are not so marked. Indeed, the combination of green buckwheat and rock phosphate resulted in a slight decrease of dry matter as compared with the combined separate increases of these materials. In the case of alfalfa there was a slight increase, and where limestone was used in addition, there was quite a marked increase. The combination of dry buckwheat and rock phosphate gave the largest increase of dry matter of all cultures over the combined separate increases of these materials, but where limestone was used in addition, there was no increase at all when calculated on this same basis.

The increases from the combination of organic matter and rock phosphate may have been due to one or more reasons. The organic matter furnished available phosphorus to the seedling and thereby promoted growth which may have enabled the plant to feed more strongly on the rock phosphate, or the rock phosphate may have been made more available by the chemical and biological processes accompanying the decay of the organic matter. However the fact that green buckwheat did not produce toxic effects and that the combination of green buckwheat and rock phosphate produced no increase over the combined separate increases of these materials, makes it questionable whether or not the organic matter increased the availability of the rock phosphate in any of the cases. The combination of alfalfa and rock phosphate which was toxic in the early stages of growth produced a marked increase only when limestone also was added, a fact which indicated that the lime prevented the toxic effect of the organic matter. The dry buckwheat com-

TABLE 6  
*The total and water-soluble phosphorus and calcium in plant tissues*

KIND OF TISSUE	TOTAL		WATER-SOLUBLE		PERCENTAGE OF TOTAL WATER-SOLUBLE		CONTENTS OF 1 TON, WATER-SOLUBLE		
	P	Ca	P	Ca	P	Ca	P	Ca	Phosphorus equivalent to that in corn crop of
	per cent	per cent	per cent	per cent	per cent	per cent	lbs.	lbs.	bu.
Buckwheat hay.....	0.318	1.544	0.262	0.080	82.1	5.2	5.24	1.6	22.5
Sweet clover hay.....	0.240	1.423	0.218	0.672	90.8	47.1	4.36	13.44	19.0
Mammoth clover hay.....	0.244	1.644	0.216	0.681	88.5	41.4	4.32	13.62	18.7
Corn stover.....	0.290	0.642	0.198	0.190	68.2	29.6	3.96	3.80	17.2
Oats straw.....	0.230	0.621	0.142	0.220	61.7	35.4	2.82	4.40	12.3

bination, which did not appear to be quite as toxic as the alfalfa combination, produced a large increase for the combination, but none when the lime was used in addition. It thus appears that the rock phosphate itself may have lessened the toxic effects produced by the organic matter. Although there is some evidence that the combination of organic matter and rock phosphate increased the availability of the latter to corn, the complication produced by the toxic effects of the organic matter does not permit definite conclusions in this respect.

In this connection the amount of water-soluble phosphorus in some of the common farm residues is of interest. In table 6 are given the amounts of total and water-soluble phosphorus and calcium in five agricultural crops. For the water-soluble phosphorus and calcium determinations, 5-gm. samples of pulverized material were shaken vigorously by hand with 500 cc. of distilled water for about 5 minutes and allowed to settle, after which the supernatant liquid was filtered in the usual way. Aliquot portions of 100 cc. were

taken for analysis. The organic matter present was oxidized with bromine water, after which the phosphorus and calcium were separated by the basic acetate method and determined in the usual manner.

The results show that 90.8 per cent of the total phosphorus in the sweet clover hay was water-soluble, while only 61.7 per cent of the total phosphorus of oats straw was water-soluble. Of the total calcium in sweet clover and buckwheat hay 47.1 per cent in the former and only 5.2 per cent in the latter was water-soluble. The soluble phosphorus in a ton of sweet clover hay is equal to the total phosphorus in 19.0 bushels of corn and correspondings stalks, while that in a ton of oats straw is equal to that in only 12.3 bushels of corn and corresponding stalks. The data of the Ohio Agricultural Experiment Station (1) indicate that alfalfa contains about the same proportion of water-soluble phosphorus as is herein reported for sweet-clover hay.

#### *The ability of plants to feed directly on rock phosphate*

In view of the fact that the return of crop residues to the soil furnishes available phosphorus to succeeding crops which may feed poorly on rock phosphate, the value of having some crops in a rotation which are able to feed strongly on rock phosphate, assumes considerable importance. It seems that the use of such crops on phosphorus-deficient soils should make possible a more effective use of rock phosphate. The value, therefore, of knowing more about the ability of various plants to feed directly on rock phosphate becomes apparent.

In most samples of rock phosphate there is a small but appreciable portion of phosphate which is readily soluble, perhaps by hydrolysis. Undoubtedly this small portion of readily soluble and available phosphate in rock phosphate is an important factor in studying the feeding power of plants for rock phosphate. This study therefore may be made in two ways. First, the rock phosphate may be used in small amounts so that the total phosphorus applied, if all available, would be just slightly more than that necessary for good plant growth. When such small amounts are used, the amount of readily available phosphate will be practically negligible as far as this study is concerned and hence the growth made by a plant will depend on its true feeding power for the kind of phosphate which makes up the greater portion of the rock phosphate. In the second place, the rock phosphate may be used in larger amounts, such as that commonly advised in field practice. With such applications much more total phosphorus is applied than is necessary for good plant growth. Obviously the amount of readily available phosphorus is thus also proportionately increased. When plants of low feeding power but possessing rather extensive and fibrous roots systems are grown under these conditions, growth will be proportionately greater than that of plants of similar feeding power, but possessing less extensive root systems, because of the ability of the former to utilize the phosphorus made available by hydrolysis. This method will

TABLE 7

*The yields of dry matter under the treatments indicated and the proportion of roots and percentage normal growth of plants grown with rock phosphate*

CROP	PART OF PLANT	TREATMENT OF PHOSPHATE AND LIME				PROPORTION OF ROOTS TO WHOLE PLANT WHEN ROCK PHOSPHATE WAS USED	PERCENTAGE NORMAL GROWTH ON ROCK PHOSPHATE COMPARED WITH ACID PHOSPHATE AT 100
		None	Acid phosphate	Rock phosphate	Rock phosphate, lime		
		gm.	gm.	gm.	gm.	per cent	per cent
Red clover.....	Tops	0.1	16.4	4.1	3.0	43.1	33.3
	Roots	0.4	5.2	3.1	2.9		
	Total	0.5	21.6	7.2	5.9		
Wheat.....	Tops	0.6	25.3	7.7	3.3	42.1	34.4
	Roots	1.3	13.4	5.6	3.0		
	Total	1.9	38.7	13.3	6.3		
Oats.....	Tops	1.0	42.0	20.1	18.8	15.9	41.5
	Roots	1.8	15.7	4.6	3.6		
	Grain	0.0	12.2	4.3	3.9		
	Total	2.8	69.9	29.0	26.3		
Corn.....	Tops	1.9	45.2	18.3		25.3	41.9
	Roots	1.2	13.3	6.2			
	Total	3.1	58.5	24.5			
Timothy.....	Tops	0.3	12.3	5.3	5.6	30.3	45.2
	Roots	0.7	4.5	2.3	3.0		
	Total	1.0	16.8	7.6	8.6		
Soybeans.....	Tops	3.6	17.2	7.3	6.4	28.1	47.7
	Roots	2.3	4.4	2.9	3.0		
	Total	5.9	21.6	10.3	9.4		
Rape.....	Tops	1.0	17.8	8.6	9.5	26.5	54.1
	Roots	0.5	3.8	3.1	5.7		
	Total	1.5	21.6	11.7	15.2		
Alfalfa.....	Tops	0.1	9.8	5.2	4.5	50.9	62.3
	Roots	0.2	7.2	5.4	4.6		
	Total	0.3	17.0	10.6	9.1		
Rye.....	Tops	0.6	15.6	7.2	6.8	64.9	66.9
	Roots	1.3	15.0	13.3	14.5		
	Total	1.9	30.6	20.5	21.3		
Buckwheat.....	Tops	1.3	14.9	10.1	4.8	17.2	72.1
	Roots	0.5	2.0	2.1	1.2		
	Total	1.8	16.9	12.2	6.0		

TABLE 7—Continued

CROP	PART OF PLANT	TREATMENT OF PHOSPHATE AND LIME				PROPORTION OF ROOTS TO WHOLE PLANT WHEN ROCK PHOSPHATE WAS USED	PERCENTAGE NORMAL GROWTH ON ROCK PHOSPHATE COMPARED WITH ACID PHOSPHATE AT 100
		None	Acid phosphate	Rock phosphate	Rock phosphate, lime		
		gm.	gm.	gm.	gm	per cent	per cent
Redtop.....	Tops	0.3	21.8	12.0	10.8	49.6	72.3
	Roots	0.7	11.1	11.8	10.3		
	Total	1.0	32.9	23.8	21.1		
Red sorrel.....	Tops	0.3	13.3	8.7	5.5	59.4	82.9
	Roots	0.4	12.5	12.7	9.8		
	Total	0.7	25.8	21.4	15.3		
Sweet clover.....	Tops	0.3	15.2	12.7	14.3	35.2	83.0
	Roots	0.8	8.4	6.9	6.4		
	Total	1.1	23.6	19.6	20.7		
Mammoth clover.....	Tops	0.3	10.5	5.0	2.4		
Alsike clover.....	Tops	0.1	6.6	3.8	2.1		

give information as to the relative order of plant growth under these conditions rather than the true feeding power of the plant. It is the method employed in the investigations reported herein.

To test the relative order of growth of various crops on rock phosphate, fifteen different kinds of plants were grown in sand cultures with rock phosphate with and without limestone. For comparison, plants were grown also without phosphorus and with acid phosphate.

The average yields of the dry matter of the tops, roots and total plants, the proportion of roots to the whole plant when grown with rock phosphate and the percentage growth made with rock phosphate compared with that made with acid phosphate, are recorded in table 7.

The results show that there was quite a wide variation in the growth of the different plants with rock phosphate used at the rate commonly advised in field practice. All the plants possessed some power to utilize the phosphorus of rock phosphate, as was indicated by the lesser growth on the check pots. Sweet clover and red sorrel produced the largest proportionate increases of dry matter and the red clover the smallest. The increases of the former were about two and one-half times as much as the latter. The use of limestone usually decreased plant growth, because of a lower availability of the phosphorus, though in a few cases it caused a slight increase in the amount of dry matter produced.

As a general rule the root systems became proportionately greater when rock phosphate was substituted for acid phosphate and still greater when limestone was used with the rock phosphate. This was evidently due to the

greater activity of the roots in trying to secure phosphorus which became increasingly more unavailable. Sweet clover, however, was an exception to this general rule. The proportion of roots to the whole plant was not definitely related to the amount of plant growth made with rock phosphate. Buckwheat and sweet clover made relatively large growths and had comparatively small root systems. Red sorrel and rye grew well and had relatively large proportions of roots. On the other hand, wheat and red clover grew poorly and had relatively high proportions of roots.

In table 8 are given the phosphorus and calcium contents of the tops and the acidity values for the leaves, stems and roots of some of the plants grown with both acid and rock phosphate.

TABLE 8  
*The phosphorus and calcium content of plant tops and acidity of the leaves, stems and roots of plants grown with acid and rock phosphates*

CROP	PHOSPHORUS CON- TENT OF PLANT GROWN WITH		CALCIUM CON- TENT OF PLANT GROWN WITH		ACIDITY OF PLANT JUICES					
	Acid phos- phate	Rock phos- phate	Acid phos- phate	Rock phos- phate	Grown with acid phosphate			Grown with rock phosphate		
					Leaves	Stems	Roots	Leaves	Stems	Roots
	per cent	per cent	per cent	per cent	pH	pH	pH	pH	pH	pH
Alfalfa.....	0.283	0.177	1.15	0.96	5.14	5.14	5.39	8.15	8.35	7.81
Buckwheat.....					5.65	4.46	4.72	5.65	3.96	4.89
Corn.....	0.135	0.083	0.57	0.56	5.36	5.36	6.04	5.36	5.67	6.02
Rape.....	0.372	0.222	1.59	1.00	5.56	5.70	5.48	5.38	5.56	5.34
Red clover.....	0.283	0.153	1.01	0.93	6.14	5.82	6.83	4.63	3.67	4.89
Redtop.....	0.301	0.206	0.63	0.42						
Rye.....					6.07	7.10	6.65	6.58	6.53	7.59
Red sorrel.....	0.403	0.311	0.94	0.59	2.93	3.63	5.82	2.79	2.99	5.51
Soybean.....	0.434	0.208	1.38	0.77	5.82	5.50	6.33	5.99	6.21	6.58
Sweet clover.....	0.310	0.314	1.37	1.36	5.75	5.82	5.97	6.49	6.53	7.17
Timothy.....	0.359	0.280	0.36	0.24						
Wheat.....					4.52	4.63	5.31	4.38	4.89	4.68

With the exception of sweet clover which gave a slight increase, all the plants analyzed gave rather large decreases in the percentage content of phosphorus when grown on rock phosphate as compared with acid phosphate. In general, neither the percentage content, nor the total amount of phosphorus in the plant tops was related to the amount of plant growth made with rock phosphate.

The calcium content of the top portions of the plants was always greater in the plants grown on acid phosphate than in those grown on rock phosphate, though in a few cases the difference was very slight. If the relation of the calcium content of the plants to the growth produced with large applications of rock phosphate is compared with the relation of the calcium content to the true feeding power of the plant for rock phosphate as enunciated by Truog



(44), it is seen that the two relations do not agree in all cases. Plants with extensive and fibrous root systems and low calcium content such as timothy and red top, produced more growth with large applications of rock phosphate than plants with somewhat higher calcium contents but less extensive root systems. This was due undoubtedly to the greater ease with which such plants were able to get the readily available portion of phosphorus from the large amounts of rock phosphate used. Red clover with a high calcium content grew but poorly on rock phosphate. In this respect the behavior of red clover, even with the larger application of rock phosphate, is similar to that obtained by Kossowitch (21) and Truog (44). In studying the feeding power of plants for rock phosphate, Truog attributed the poor showing of red clover to the extremely small seeds which were unable to furnish the seedling with sufficient phosphorus for a good start. He was able to demonstrate that alfalfa and tobacco, both small-seeded plants of high calcium content, became much better feeders after the root systems were more thoroughly developed. Of the cultures reported herein, sweet clover, mammoth clover, and alsike clover, all small-seeded plants of high calcium content, grew much better on rock phosphate than red clover, a fact which suggests the possibility of other under-terminated factors. It was observed, however, that of all the plants grown, red clover produced the largest increase in the proportion of roots when rock phosphate replaced acid phosphate, and also the largest increase when limestone was used with the rock phosphate. The acidity of the juices of red clover grown on rock phosphate was one of the highest from all of the plants tested. The acidity of the juice of red clover grown on acid phosphate was slight.

The acidity values of the juice of the leaves, stems and roots varied considerably with the different plants. In general the juices of the plants were more acid when grown with acid phosphate than with rock phosphate. Wheat and red clover, however, were more acid when grown with rock phosphate, a fact which may be related to the poor growth of these plants with rock phosphate. On the whole, there was no relation of the acidity of the plants to their ability to feed directly on rock phosphate. Red sorrel and sweet clover both grew well on rock phosphate; the former was high in acidity, while the latter was low.

In connection with the theory that plants high in calcium are good feeders on rock phosphate, it was thought that the testing of the feeding powers of a few typical plants for the potash in feldspar, in which case the removal of calcium is not a factor, would be of interest. For this test, corn, oats, sweet clover and buckwheat were grown in sand cultures with rock phosphate. Finely powdered feldspar at the rate of 2 tons to the acre was used as the source of potassium. The nutrient solution was modified from that used in the phosphate studies by substituting mono-calcium phosphate for the potassium sulfate. Checks without potash and with potassium sulfate were used for comparison. The sand used was previously leached to remove any soluble



potassium. The work was done in duplicate. The growth of the plants with the soluble potash was not so good as desired. Of the four crops, sweet clover grew the nearest to normal. The reason for the lack of vigor of the other crops is not known. This condition made the feeding powers of the plants on feldspar appear abnormally high. The crops were harvested 50 to 60 days from the time of seeding, and the results are recorded in table 9.

The oats and sweet clover grew as well on the feldspar as on the soluble potash. Corn and buckwheat, however, did very poorly on the feldspar. The chief differences between the feeding power of these plants for feldspar and rock phosphate is in the position of oats and buckwheat. With rock phosphate buckwheat produced a large growth; with feldspar it scarcely

TABLE 9

*The growth of plants in quartz cultures with no potash, soluble potash and feldspar potash*

KIND OF PLANT	PART OF PLANT	YIELDS OF DRY MATTER WITH TREATMENTS AS FOLLOWS			NORMAL GROWTH ON FELDSPAR
		None	Soluble potash	Feldspar	
		gm.	gm.	gm.	per cent
Buckwheat.....	Tops	1.0	7.4	1.6	23.3
	Roots	0.5	2.0	0.6	
	Total	1.5	9.4	2.2	
Corn.....	Tops	5.0	26.5	9.5	45.2
	Roots	2.5	4.5	4.5	
	Total	7.5	31.0	14.0	
Sweet clover.....	Tops	2.5	5.4	5.4	106.5
	Roots	1.0	2.2	2.7	
	Total	3.5	7.6	8.1	
Oats.....	Tops	5.0	8.3	7.7	107.6
	Roots	1.2	2.2	3.6	
	Total	6.2	10.5	11.3	

grew. Oats showed the opposite condition, though not to such a marked extent. The only explanation that can be given for this reversal in the growth of these plants is the difference in the character of the root systems of the two crops. Buckwheat has a scanty root system. Oats has an extensive and fibrous root system, making it possible for the oats to absorb the soluble potassium produced by hydrolysis or otherwise, much more completely than buckwheat. Although the results with feldspar are somewhat irregular, an important point is indicated. Buckwheat feeds poorly on feldspar, but strongly on rock phosphate. The reason for this reversal is found in the theory that plants high in calcium are strong feeders on rock phosphate. In the case of feldspar there is no soluble calcium to remove, and the ability to utilize the soluble potassium is probably determined largely by the extent

of the root system. The oat plant has an extensive root system and thus feeds strongly on it.

Of all the plants grown in sand cultures with rock phosphate and feldspar sweet clover showed the most remarkable feeding power. To demonstrate further the feeding power of sweet clover for relatively insoluble plant-food materials, inoculated seeds were planted in pure quartz sand in 2-gallon pots. Rock phosphate was added at the rate of 1 ton, and dolomitic limestone and feldspar each at the rate of 2 tons to the acre. Distilled water was used to water the plants. The first application of water contained the regular amount of  $\text{NaNO}_3$  used in the pots-culture studies previously reported. In the course of time a fairly luxuriant growth of sweet clover resulted. A similar experiment with corn resulted in only a very small growth with the production of tassels when the plants were about 12 inches high.

Just why sweet clover is such a good feeder is not entirely clear. The fact that it is high in calcium aids in the explanation of its feeding powers for rock phosphate. However, this explanation will not hold for its high feeding power for feldspar. There are evidently other factors which are important in this connection.

In addition to its strong feeding powers, sweet clover is a crop which lends itself well to rotation schemes. It may be seeded in small grain and used for pasture and hay purposes the next season, or plowed down in the preparation of corn land the following spring. The last method is especially well adapted to grain systems of farming for the effective use of rock phosphate. Undoubtedly there are many ways in which the farmer could advantageously use the strong feeding powers of plants like sweet clover, rape and buckwheat.

#### SUMMARY

It is commonly believed that organic acids, carbon dioxide and nitrous acid resulting from the decay of organic matter are active agents in making rock phosphate available to plants. When the effect of organic matter on the availability of rock phosphate has been studied in the laboratory, increases of water- or citrate-soluble phosphorus have rarely been found. These laboratory experiments have usually been made without provision for the removal of the phosphorus and calcium as made soluble, as is the case under field conditions. For this reason it seemed highly desirable to conduct further studies on this problem. Experiments were planned in which common forms of organic matter were used with rock phosphate. In some of these experiments provision was made for the removal of the soluble phosphorus and calcium in order more nearly to imitate field conditions in this respect.

Experiments were made as follows: (a) rock phosphate was mixed with several forms of organic matter in sand and soil with no provision for the removal of phosphorus as it became soluble; (b) in similar mixtures provision was made by leaching for the removal of phosphorus as it became soluble;

(c) in still other similar mixtures provision was made by upward-moving capillary water for the removal of phosphorus as it became soluble; (d) in a fourth experiment the solvent effect of organic-matter extracts with and without carbon dioxide on rock phosphate was studied; (e) pot culture experiments also were made in this problem, and in addition the feeding powers of plants in relation to the utilization of rock phosphate and feldspar were investigated. The results are summarized as follows:

1. Experiments (a), (b), (c) and (d) failed to show a solvent effect of the decaying organic matter on rock phosphate; and in accord with the results of several other investigators the results in all cases showed a decreased amount of soluble phosphorus from that present at the beginning.

2. The failure of the experiments to show an increase in the availability of rock phosphate was probably due to the fact that when organic matter such as was used, decomposes, sufficient bases are liberated along with the acids that are formed, to neutralize the acids and prevent their action on rock phosphate. Some of the soluble phosphorus possibly was used by bacteria, as indicated by Tottingham and Hoffman (41). When mixtures of rock phosphate and organic matter are applied to soils, the conditions are different, because of the capacity of soils to take up basic material, especially if they are acid.

3. In some cases mixtures of organic matter and rock phosphate applied to pot cultures produced increases in the growth of corn over the combined separate increases of organic matter and rock phosphate. The phosphorus of the organic matter became available readily to corn. The decaying organic matter either had a solvent action on the rock phosphate, or else the phosphorus supplied by the organic matter gave the corn seedlings a better start, so that they were enabled to exert a stronger feeding power toward the rock phosphate.

4. A study of the growth of fifteen different plants on rock phosphate in sand cultures showed a wide variation in the amount of dry matter produced. There was no definite relation of the amount of plant growth to the extent of root systems, the phosphorus content, or the acidity of the plant juices. The applications of rock phosphate were too large to allow a critical examination of the relation between the feeding power and the calcium content.

5. The greater ability of some plants which are high in calcium to feed on rock phosphate than on feldspar is in accord with the theory that plants high in calcium are good feeders on rock phosphate.

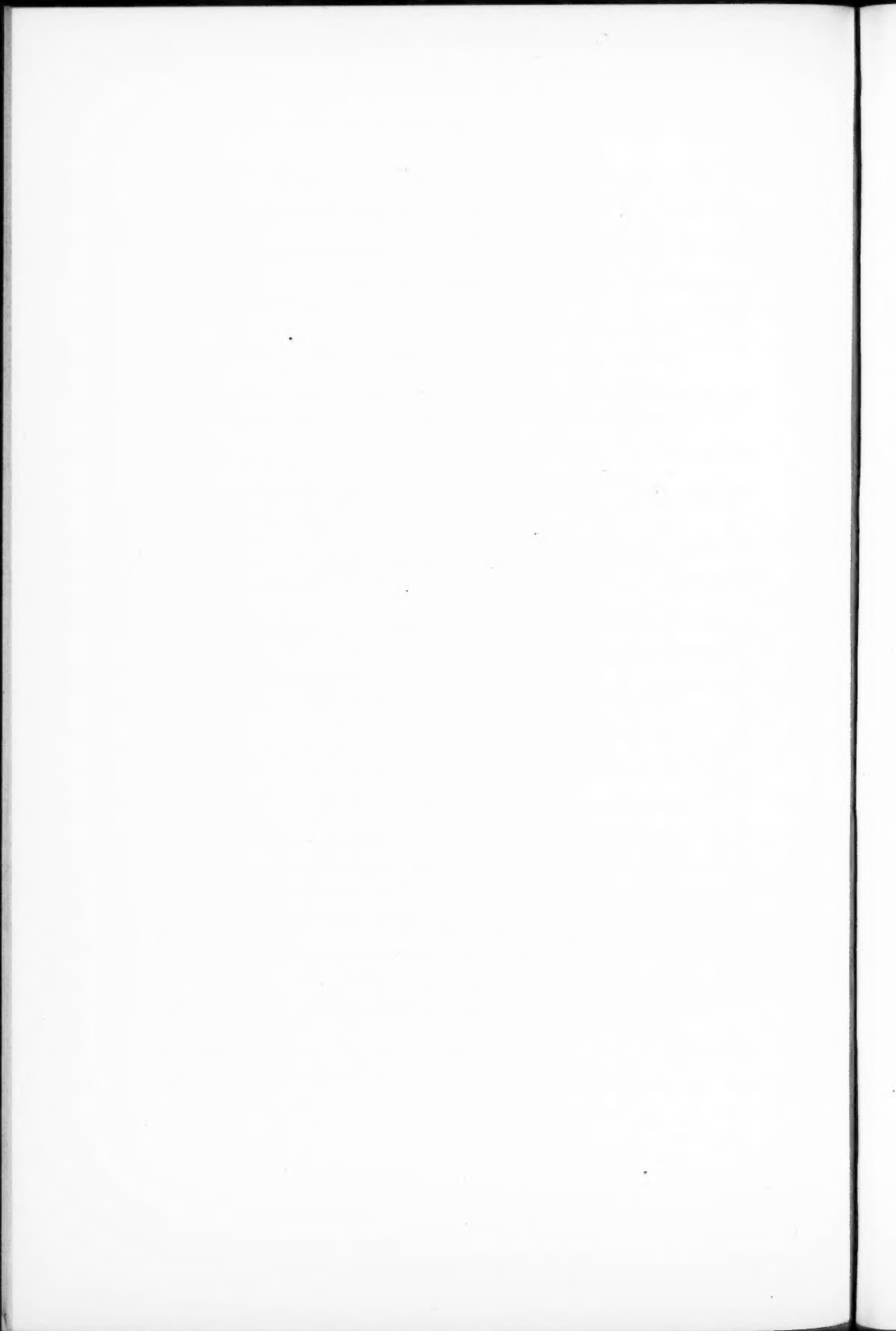
6. Sweet clover possesses remarkable feeding powers toward rock phosphate and feldspar and is well suited to rotations for the utilization of these.

7. The possibility of growing crops of high feeding power to supply organic matter and available phosphorus in rotations with crops of low feeding power is a question of considerable practical importance in the utilization of rock phosphate.

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## EXPERIMENTS IN THE TREATMENT OF BALLED EARTH ABOUT THE ROOTS OF CONIFEROUS PLANTS FOR THE CONTROL OF JAPANESE BEETLE LARVAE

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### INTRODUCTION

A Federal plant quarantine in operation during the spring of 1920 prohibited the interstate shipment of soil or coniferous plants with soil about their roots from within a restricted area in New Jersey infected by the Japanese beetle (*Popillia japonica*). As a result of this quarantine two nurseries engaged in growing coniferous and certain other ornamental stock such as azaleas, were restricted in the marketing of their product, a condition rendered necessary by the fact that no safe method exists for the killing of insects which may be contained within the soil about the roots of plants. Under these circumstances a series of experiments were conducted during 1920 at Riverton, N. J. to determine the comparative value of a certain class of *gas-producing* compounds as killing agents against the soil-inhabiting Japanese beetle grub and also the effect of these toxic compounds upon the plants concerned.

### NURSERY PRACTICE IN SHIPPING CONIFEROUS PLANTS

In order that coniferous plants, such as *Arbor vitae*, spruces, pines, etc., may be successfully transplanted, it is essential that the tree be dug in such a way that a sufficient proportion of the roots and rootlets are not disarranged in the soil or exposed to the drying action of the atmosphere. Figure 1 of plate 1 illustrates a small conifer which has been carefully dug from the nursery row so as to preserve the roots within the soil-ball. In order to secure a good soil-ball about the roots nurserymen transplant conifers at frequent intervals in the nursery row. This frequent transplanting with the accompanying root pruning, results in a compact mass of fibrous roots in the immediate vicinity of the base of the tree. This compact mass of roots and soil, or or the *soil-ball* as it is called, facilitates handling and insures successful transplanting. As an added precaution in shipping this class of stock the soil-ball is wrapped in burlap (pl. 1, fig. 2).



## POSSIBILITY OF INFESTATION OF SOIL-BALLS BY INSECTS; THE LONG LARVAL PERIOD OF THE JAPANESE BEETLE

The possibility of these soil-balls being infested with soil-inhabiting animals such as root aphids, wire-worms, white grubs, nematodes, etc., has been confirmed by the finding of various specimens in the loose soil in the bottom and sides of the holes from which balled plants had been removed. An examination of a block of azaleas in one of the local nurseries disclosed the presence of larvae of the Japanese beetle in among the plants at one end of the block, the infestation no doubt resulting from the fact that the rows at that point were somewhat weedy, a condition favorable to the deposition of eggs by the Japanese beetle. In this connection it is well to remember that the larvae of *Popillia japonica* are found in the soil throughout the year, young grubs just hatched from the eggs being present in the soil before all the old grubs have pupated and emerged as beetles. There therefore exists no fixed period in the year within which balled plants could be shipped with the absolute assurance that no grubs would be contained within the shipment.

## BASIS OF THE EXPERIMENTAL WORK

As stated above, this class of nursery stock when ready for shipment is tightly wrapped in burlap bagging. Under these circumstances the plants can be dipped or soaked in solutions with impunity as far as mechanical injury is concerned, since only a very small proportion of the soil escapes through the bagging and the relative position of the roots within the soil-ball is undisturbed. On removal from the solution the plants would be ready for shipment after being allowed to drain for a few hours.

## CLASSES OF COMPOUNDS EMPLOYED

The following classes of compounds were employed dissolved in water.

- I. Gas-producing compounds soluble in water.
  - a. Such compounds slightly soluble in water, e.g., carbon disulfide.
  - b. Such compounds readily soluble in water, e.g., sodium sulfocarbonate.
- II. Corrosive compounds, e.g., copper sulfate.

## EXPERIMENTAL PROCEDURE

Aside from the few initial experiments the following steps were taken in testing the various compounds:

I. *Popillia* grubs were dipped in solutions of varying concentrations of the material being tested for varying periods of time. Throughout this set of dipping tests the solutions were maintained at a temperature of approximately 70°F.

II. If a compound gave evidence of positive value as a killing agent when used as above a second series of experiments were made with the material.

In these latter experiments the grubs, *embedded in soil-balls*, were subjected to the action of the material in solution.

III. The material in solution was tested as to its action on the plant, small coniferous plants with balled earth about their roots being employed.

#### DIPPING TESTS WITH GRUBS OF *POPILLIA JAPONICA*

In making these tests 1,000 cc. of the solution was placed in a glass beaker. The grubs were prevented from sinking to the bottom of the beaker by means of a muslin or wire screen, the object being to retain the grubs in the exact vertical center of the solution. The results of these dipping tests are given in tables 1, 2, and 3.

#### *Sodium sulfocarbonate*

*Preparation.* This material was prepared from powdered fused sodium sulfide and carbon disulfide by agitation. The sodium monosulfide ( $\text{Na}_2\text{S}$ ) deliquescent crystals were melted, the liquid poured into a shallow pan, allowed to harden and ground to a powder in a mortar. A quantity of this powder was then placed in a mason jar with twice the volume of carbon disulfide and agitated at short intervals for 2 hours. If larger quantities are desired a kitchen butter-churn lends itself to this purpose. The resulting sodium sulfocarbonate is heavier than either the excess carbon disulfide or sludge of impurities remaining after the chemical action is complete. On standing for a short time the excess carbon disulfide rose to the top of the mixture and was decanted. The remaining mixture of sodium sulfocarbonate and sludge was then filtered, the sludge remained on the filter-paper while the filtrate consisted of sodium sulfocarbonate.

Carbon disulfide acts as an acid toward alkaline sulfides. The reaction with sodium sulfide is as follows:



*Properties.* The sodium sulfocarbonate resulting from the above method of preparation is a heavy, reddish liquid and tests about 45° Baumé. It is soluble in water in all proportions. The compound is unstable and reacts with water and carbon dioxide in the air or soil. On decomposing, it yields about 20 per cent carbon disulfide and 9 per cent hydrogen sulfide (1). The reaction is as follows:



Organic acids produce the same result. The reaction with acetic acid is as follows:



Table 1 gives the results of dipping *Popillia* grubs in various solutions of sodium sulfocarbonate, the grubs *not* embedded in soil. When grubs were

dipped in a solution of 15 cc. of sodium sulfocarbonate per gallon for 220 minutes, 100 per cent mortality resulted. On the other hand, when grubs were dipped in a solution of 15 cc. of sodium sulfocarbonate and 15 cc. of acetic acid per gallon the grubs were all dead in 30 minutes. These results indicate that while sodium sulfocarbonate itself has some killing ability the active killing agent is the carbon disulfide resulting from the decomposition of the sodium sulfocarbonate by organic acids.

TABLE 1

*Results of dipping Popillia grubs in solutions of sodium sulfocarbonate; grubs in the third (last instar\* and not in soil*

CONCENTRATION OF SOLUTION	DURATION OF DIPPING	Kill
	min.	per cent
30 cc. sodium sulfocarbonate per gallon.....	30	33
30 cc. sodium sulfocarbonate per gallon.....	60	33
30 cc. sodium sulfocarbonate per gallon.....	90	66
30 cc. sodium sulfocarbonate per gallon.....	120-230	100
15 cc. sodium sulfocarbonate per gallon.....	30	0
15 cc. sodium sulfocarbonate per gallon.....	60	33
15 cc. sodium sulfocarbonate per gallon.....	120	66
15 cc. sodium sulfocarbonate per gallon.....	220-270	100
15 cc. sodium sulfocarbonate plus 15 cc. 36 per cent acetic acid per gallon.....	15	66
15 cc. sodium sulfocarbonate plus 15 cc. 36 per cent acetic acid per gallon.....	30-120	100
30 cc. sodium sulfocarbonate plus 15 cc. 36 per cent acetic acid per gallon.....	15	0
30 cc. sodium sulfocarbonate plus 15 cc. 36 per cent acetic acid per gallon.....	30	33
30 cc. sodium sulfocarbonate plus 15 cc. 36 per cent acetic acid per gallon.....	60-120	100
30 cc. sodium sulfocarbonate plus 30 cc. 36 per cent acetic acid per gallon.....	15	100
30 cc. sodium sulfocarbonate plus 30 cc. 36 per cent acetic acid per gallon.....	30-120	100
Water only (checks).....	15-300	0

\* Tests were made using first and second instar grubs and pupae of *Popillia japonica*. The tests indicate that the first and second instar grubs are only slightly less resistant to toxic compounds than third instar grubs while the pupae are equal to the third instar grub in resistance.

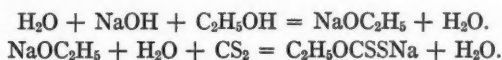
#### *Sodium ethyl xanthate*

*Preparation and properties.* Sodium ethyl xanthate<sup>1</sup> was made as follows: 200 gm. of sodium hydroxide was dissolved in 200 cc. of water and the mixture cooled. To this was added 300 cc. of alcohol and the mixture shaken. Then 310 cc. of carbon disulfide was added in small portions, with shaking and cooling after each addition to prevent loss of CS<sub>2</sub>.

<sup>1</sup> The writers are indebted to Dr. C. C. McDonnell, Insecticide and Fungicide Board, U. S. D. A., for the preparation of this material.

which would occur if the flask were allowed to heat above approximately 45°. A slight excess of carbon disulfide and alcohol was used in order to get as nearly as possible to a neutral reaction. The product obtained should contain about 70 per cent of the crude xanthate. This product is of course not as pure as that made under anhydrous conditions.

The reactions are as follows:



Sodium ethyl xanthate is orange yellow in color and is readily soluble in water. Decomposition of the aqueous solution into carbon disulfide, ethyl

TABLE 2

*Results of dipping Popillia grubs in solutions of sodium ethyl xanthate in water; grubs in the third (last) instar, and not in soil*

CONCENTRATION OF SOLUTION	DURATION OF DIPPING	KILL
	hours	per cent
0.5 per cent.....	1-3	0
1.0 per cent.....	1-3	0
1.5 per cent.....	1-3	0
0.125 per cent + 0.16 per cent acid*.....	1-3	0
0.25 per cent + 0.16 per cent acid.....	1	0
0.25 per cent + 0.16 per cent acid.....	2-3	33
0.25 per cent + 0.33 per cent acid.....	1	0
0.25 per cent + 0.33 per cent acid.....	2	33
0.25 per cent + 0.66 per cent acid.....	1	0
0.25 per cent + 0.66 per cent acid.....	2	100
0.25 per cent + 1 per cent acid.....	1	0
0.25 per cent + 1 per cent acid.....	2	100
0.5 per cent + 0.125 per cent acid.....	1	0
0.5 per cent + 0.125 per cent acid.....	2-3	100
0.5 per cent + 0.16 per cent acid.....	1-2	33
0.5 per cent + 0.16 per cent acid.....	3	100
0.5 per cent + 0.33 per cent acid.....	1	33
0.5 per cent + 0.33 per cent acid.....	2	100
0.5 per cent + 0.50 per cent acid.....	1	33
0.5 per cent + 0.50 per cent acid.....	2	100
0.5 per cent + 0.66 per cent acid.....	1	0
0.5 per cent + 0.66 per cent acid.....	2	100
0.5 per cent + 1 per cent acid.....	1	66
0.5 per cent + 1 per cent acid.....	2	100
1.0 per cent + 0.125 per cent acid.....	1	33
1.0 per cent + 0.125 per cent acid.....	2-3	100
1.0 per cent + 0.33 per cent acid.....	1-2	100
1.0 per cent + 0.66 per cent acid.....	1-2	100
1.0 per cent + 1 per cent acid.....	1-2	100
Water only (checks).....	1-3	0

\* 36 per cent acetic acid.

alcohol and sodium hydroxide begins at 77°F. and is completed on boiling. The reaction is as follows:



The aqueous solution is decomposed without heating by acetic acid as follows:



The pure xanthate contains about 47 per cent carbon disulfide.

Table 2 gives the results of dipping *Popillia* grubs in various solutions of sodium ethyl xanthate.

It will be noted from table 2 that a 1.5 per cent solution of sodium ethyl xanthate (with no acid added) failed to kill *Popillia* grubs in 3 hours whereas a solution of 0.25 per cent xanthate plus 0.66 $\frac{2}{3}$  per cent acetic acid killed the grubs in 2 hours. This difference in killing properties between the xanthate alone and the xanthate plus acid is due to the fact that the xanthate without acid does not give off carbon disulfide except at higher temperatures, whereas with the xanthate plus acid combination the compound breaks up into carbon disulfide and alcohol and the resulting carbon disulfide kills the grubs.

#### Emulsions

Several mixtures of turpentine, castor oil, carbon disulfide and soap were compounded in various proportions for the purpose of obtaining an emulsion which would hold the carbon disulfide in suspension indefinitely. A good emulsion of this sort can be made according to the following formula: dissolve 4 cc. of carbon disulfide in 4 cc. of castor oil; add 4 cc. of turpentine; add 16 cc. of soap solution ( $\frac{1}{2}$  pound of hard soap dissolved in 1 gallon of water). This emulsion did not separate into its component parts to the slightest degree at the end of 2 months. When the emulsion was added to water it remained in suspension for hours before the carbon disulfide began to settle to the bottom of the container.

Table 3 gives the results of dipping tests with this emulsion used at various strengths. It required a 2-hour exposure to a 1 per cent emulsion to give a 100 per cent kill, while the same results were obtained by a  $\frac{1}{2}$ -hour exposure to a 2 per cent emulsion. The killing properties of this emulsion are no doubt due to the carbon disulfide contained therein.

#### Miscellaneous dipping tests

Table 3 gives the results of dipping *Popillia* grubs in aqueous solutions of various compounds.

It will be observed that such compounds as copper sulfate, potassium fluoride and nicotine, all practically non-volatile compounds at ordinary temperatures, had a negligible effect on *Popillia* grubs. On the other hand, volatile compounds, the vapors of which are but slightly soluble in water,

such as carbon disulfide, thymol<sup>2</sup> and mustard oil, were decidedly toxic to the grubs.

The four sets of dipping tests detailed above indicate that *the only class of compounds in solution toxic to Popillia grubs are those capable of producing a*

TABLE 3

*Results of dipping Popillia grubs in solutions of various compounds; grubs in the third (last) instar, and not embedded in soil*

CONCENTRATION OF SOLUTION	DURATION OF DIPPING	KILL
		per cent
3.5 gm. sodium cyanide per gallon.....	10-110 min.	0
5.0 per cent sodium chloride.....	1-15 hrs.	0
5.0 per cent copper sulfate.....	15-30 min.	0
5.0 per cent copper sulfate.....	1 hr.	33
5.0 per cent copper sulfate.....	2 hrs.	66
5.0 per cent copper sulfate.....	3 hrs.	33
5.0 per cent copper sulfate.....	18 hrs.	0
5.0 per cent copper sulfate.....	24 hrs.	0
0.16 per cent nicotine.....	1-3 hrs.	0
1.0 per cent potassium fluoride.....	1-2 hrs.	0
5.0 per cent potassium fluoride.....	1-2 hrs.	0
10.0 per cent potassium fluoride.....	1-2 hrs.	0
Camphor (saturated solution).....	1-2 hrs.	0
Carbon disulfide (saturated solution)*.....	15 min.	33
Carbon disulfide (saturated solution)*.....	30-120 min.	100
Thymol (saturated solution)†.....	1-2 hrs.	100
Mustard oil (saturated solution)‡.....	15 min.	66
Mustard oil (saturated solution)‡.....	30-60 min.	100
Chloroform (saturated solution).....	15-45 min.	100
5 per cent of carbon disulfide emulsion§.....	1-2 hrs.	0
1 per cent of carbon disulfide emulsion§.....	2 hrs.	100
2 per cent of carbon disulfide emulsion§.....	½-1 hr.	100
3 per cent of carbon disulfide emulsion§.....	½-1 hr.	100
Water only (checks).....	1-24 hrs.	0

\* Approximately one part carbon disulfide in 1,000 parts water.

† See footnote below

‡ Allyl isothiocyanate (artificial), page 48.

§ See page 50.

gas insoluble or only slightly soluble in water. Substances such as copper sulfate, which in a 5 per cent solution reacts quickly on iron wire, has no appreciable effect on the grub.

<sup>2</sup> Thymol—a phenol found in volatile oil of thymus sp.; colorless, translucent, crystalline, 1 part soluble in 1,100 parts of water.

Mustard oil (allyl isothiocyanate, artificial). For a report of the above compounds when used as anthelmintics see Sollmann (3).

*Dipping tests with grubs of Popillia japonica embedded in soil-balls*

Plate 2 illustrates the apparatus employed in testing the various materials dissolved in water against the larvae in soil. The small wire cages shown in figure 4 were made of wire screen, (16 meshes to the inch), and were  $1\frac{3}{4}$  inches long and  $\frac{1}{2}$  inch in diameter. Small cork stoppers, readily removable, were used to plug the ends. In using these cages a bit of soil was placed in the cage (holding it upright), then a healthy grub placed within, the cage filled with soil and stoppered. By maintaining the soil in a moist condition grubs could be kept in a normal condition in these small cages for many days or until they became restive and gnawed their way out through one of the cork stoppers.

Figure 1, plate 2, illustrates a wire cylinder 8 inches in diameter and 10 inches in height. Figure 2, plate 2 illustrates the same cylinder covered with a double thickness of muslin.

*The object in using these two types of containers was to simulate as closely as possible the natural soil-ball.* This subterfuge is necessary because it is impossible to obtain naturally the exact experimental condition required in this sort of work. The balled earth about the roots of a coniferous plant might or might not contain grubs and it is obvious that any examination made to determine the presence of grubs in such a soil-ball and their exact location therein would so disarrange the ball as to render it unfit for experimental purposes.

Under these circumstances it was necessary to resort to the use of artificial soil-balls. Such a ball was manufactured as follows. Soil was placed in the bottom of the cloth-covered cylinder and tamped down firmly to a depth of  $\frac{1}{2}$  inch. Three of the small wire cages described above, each containing a healthy grub, were then placed on top of the half-inch of soil in the large cylinder, one in the center and two within  $\frac{1}{2}$  inch of the sides. More soil was placed in the cloth-covered cylinder and tamped down to a total depth of  $3\frac{3}{4}$  inches and another three grubs in small cages introduced as before. Again more soil to a total depth of  $7\frac{1}{2}$  inches was placed in the large cylinder and a third and final set of three grubs placed thereon and covered with  $\frac{1}{2}$  inch of soil and tamped down.

This arrangement gave a soil "ball" 8 inches in diameter and 8 inches in height containing 9 grubs, one in the center of the ball and eight distributed around the top, sides and bottom of the ball within  $\frac{1}{2}$  inch of the surface. By confining the grubs in these small wire cages they were held in the exact locations desired within the soil-ball while the muslin cover of the cylinder was comparable to the burlap bagging used in wrapping the soil-ball of a coniferous plant. In running a series of tests with a material it was a simple matter to make the required number of these soil-balls and to dip them, entirely submerged, in solutions contained in galvanized iron buckets or tubs.

The soil used throughout these tests consisted of a light sandy or garden loam. In these soil-ball tests the only soil condition which it seemed advis-



able to avoid—as indicated by the results—was excessively wet soil. Throughout this set of experiments the temperature of the soil-ball and of the solutions was recorded. These temperature records indicate the inadvisability of

TABLE 4

*Results of dipping Popillia grubs, embedded in soil-balls, in aqueous solutions of sodium sulfocarbonate; grubs in third (last) instar*

AMOUNT OF MATERIAL PER GALLON	DURATION OF DIPPING	KILL	SOIL TEMPERATURE	SOLUTION TEMPERATURE
		per cent	°F.	°F.
15 cc. sodium sulfocarbonate.....	30 min.	66.6	65-75†	85-79‡
15 cc. sodium sulfocarbonate.....	60 min.	88.9	64-79	74-71
15 cc. sodium sulfocarbonate.....	120 min.	66.6	63-70	75-71
30 cc. sodium sulfocarbonate.....	15 min.	66.6	57-63	85-77
30 cc. sodium sulfocarbonate.....	30 min.	100.0	67-74	83-79
30 cc. sodium sulfocarbonate.....	60 min.	100.0	61-67	75-68
30 cc. sodium sulfocarbonate.....	120 min.	100.0	61-67	75-68
5 cc. sodium sulfocarbonate plus 20 cc. sodium carbonate.....	60 min.	22.2	79§	75¶
10 cc. sodium sulfocarbonate plus 20 cc. sodium carbonate.....	60 min.	55.5	80	75
15 cc. sodium sulfocarbonate plus 20 cc. sodium carbonate.....	60 min.	55.5	80	75
30 cc. sodium sulfocarbonate plus 20 cc. sodium carbonate.....	60 min.	88.9	80	75
5 cc. sodium sulfocarbonate in H <sub>2</sub> O charged with CO <sub>2</sub> .....	60 min.	0	68-69	76-75
10 cc. sodium sulfocarbonate in H <sub>2</sub> O charged with CO <sub>2</sub> .....	60 min.	66.0	68-69	76-75
15 cc. sodium sulfocarbonate in H <sub>2</sub> O charged with CO <sub>2</sub> .....	60 min.	88.9	68-69	76-75
5 cc. sodium sulfocarbonate plus 8 cc. acetic acid*....	60 min.	11.1	63-73	76-73
10 cc. sodium sulfocarbonate plus 8 cc. acetic acid*....	60 min.	33.3	63-73	76-73
15 cc. sodium sulfocarbonate plus 8 cc. acetic acid*....	60 min.	33.3	63-73	76-73
10 cc. sodium sulfocarbonate plus 10 cc. acetic acid....	24 hrs.	0	81	65
10 cc. sodium sulfocarbonate plus 15 cc. acetic acid....	1 hr.	55.5	71	75
20 cc. sodium sulfocarbonate plus 20 cc. acetic acid....	1 hr.	66.0	71	75
25 cc. sodium sulfocarbonate plus 25 cc. acetic acid....	1 hr.	88.9	71	75
Water only (check).....	1-24 hrs.	0	As above	As above

\* 36 per cent acetic acid.

† Initial temperature of soil ball 65° F., raised to 75° as a result of dipping in solution with initial temperature of 85°.

‡ Initial temperature of solution 85° F., reduced to 79° by cooling effect of soil-ball with initial temperature of 65°.

§ Initial temperature of soil-ball.

¶ Initial temperature of solution.

allowing the soil or dip to run below 60° while 70° to 75° is a good working temperature from the standpoint of both results and ease in handling materials.

The results of these dipping tests with soil-balls containing *Popillia* grubs are given in tables 4 and 5. The dipping of soil-balls in a solution of 30 cc. of sodium sulfocarbonate to the gallon of water for a period of 30 minutes

gave 100 per cent control; the grubs were dead at the end of 5 days, during which time the soil-ball had remained undisturbed at a room temperature of 70°F. During these 5 days the sodium sulfocarbonate taken up by the soil-ball while in the solution reacted with the carbon dioxide in the soil, giving off carbon disulfide in sufficient quantity to produce 100 per cent mortality.

The dipping of soil-balls in a solution containing 30 cc. of sodium sulfocarbonate and 20 gm. of sodium carbonate per gallon did not give 100 per

TABLE 5

*Results of dipping Popillia grubs, embedded in soil-balls, in aqueous solutions of various compounds; grubs in third (last) instar*

SOLUTION	DURATION OF DIPPING	KILL	SOIL TEMPER- ATURE	SOLUTION TEMPER- ATURE
		per cent	°F.	°F.
0.25 per cent sodium ethyl xanthate.....	24 hrs.	11.1	80†	78‡
1.5 per cent sodium ethyl xanthate.....	1 hr.	77.8	80	78
0.5 per cent sodium ethyl xanthate plus 0.5 per cent acetic acid*.....	1-2 hrs.	66.0	73	69
1 per cent sodium ethyl xanthate plus 1 per cent acetic acid*.....	1 hr.	100.0	73	69
1.5 per cent sodium ethyl xanthate plus 1.5 per cent acetic acid*.....	1 hr.	100.0	73	69
Saturated solution of hydrogen sulfide.....	1 hr.	33.3	68	75
Saturated solution of carbon disulfide.....	15 min.	11.1	80	75
Saturated solution of carbon disulfide.....	60 min.	0	79	75
Saturated solution of carbon disulfide.....	120 min.	0	79	75
Saturated solution of carbon disulfide.....	24 hrs.	0	81	75
Saturated solution of CS <sub>2</sub> and H <sub>2</sub> S.....	1 hr.	0	60	75
2 per cent "solution" of carbon disulfide emulsion....	1 hr.	44.4	79	75
3 per cent "solution" of carbon disulfide emulsion....	1 hr.	33.3	79	75
Thymol (saturated solution).....	1 hr.	22.3	79	78
Thymol (saturated solution).....	6 hrs.	33.3	79	78
Thymol (saturated solution).....	12 hrs.	22.3	79	78
Thymol (saturated solution).....	24 hrs.	11.1	79	78
Mustard oil (saturated solution).....	24 hrs.	66.6	79	78
Saturated solution of mustard oil and CS <sub>2</sub> .....	24 hrs.	66.6	79	78
Water only (checks).....	1-24 hrs.	0	As above	As above

\* 36 per cent acetic acid.

† Temperature of soil-ball before dipping.

‡ Temperature of solution at beginning of dipping period.

cent mortality when examined 5 days later. This is explained by the fact that the sodium carbonate reacted with any carbon dioxide present in the soil (forming sodium bicarbonate,  $\text{NaHCO}_3$ , viz.:  $\text{Na}_2\text{CO}_3 + \text{H}_2\text{O} + \text{CO}_2 = 2\text{NaHCO}_3$ ) thereby preventing the decomposition of the sodium sulfocarbonate into carbon disulfide.

Nothing apparently was gained by the addition of acetic acid to solutions of sodium sulfocarbonate employed in dipping soil-balls containing *Popillia*

grubs; acetic acid caused a much more rapid decomposition of the sulfocarbonate with consequent evolution of carbon disulfide, but the same mortality was secured by using an equal concentration of sulfocarbonate without acid, indicating that the carbon dioxide occurring naturally in the soil is sufficient in quantity to decompose the sulfocarbonate.

Table 5 gives the results of dipping *Popillia* grubs, embedded in soil-balls in aqueous solutions of various compounds. The dipping of soil-balls in solutions of sodium ethyl xanthate gave an indifferent grub mortality due to the fact that the material was not apparently acted upon by the weak concentration of carbonic acid in the soil. A combined solution of 1 per cent xanthate and 1 per cent acetic acid gave 100 per cent mortality. In this case the carbon disulfide formed by the reaction of the acid and the xanthate was the active killing agent.

TABLE 6

*Comparison of results obtained in dipping Popillia grubs embedded and not embedded in soil, in solutions of various compounds*

SOLUTION	POPILLIA GRUBS NOT EMBEDDED IN SOIL		POPILLIA GRUBS EMBEDDED IN SOIL-BALLS	
	Duration of dipping	Kill	Duration of dipping	Kill
		per cent		per cent
0.5 per cent sodium ethyl xanthate plus 0.5 per cent of 36 per cent acetic acid.....	2 hrs	100	2 hrs.	66.0
15 cc. sodium sulfocarbonate plus 15 cc. of 36 per cent acetic acid per gallon.....	30 min.	100	1 hr.	55.5
Carbon disulfide (saturated solution).....	30 min.	100	24 hrs.	0
2 per cent of carbon disulfide emulsion.....	30 min.	100	1 hr.	33.0
Saturated solution of thymol.....	60 min.	100	24 hrs.	11.1
Mustard oil (saturated solution).....	30 min.	100	24 hrs.	66.6

The dipping of soil-balls containing *Popillia* grubs in saturated aqueous solutions of carbon disulfide, thymol and mustard oil and a 3 per cent solution of carbon disulfide emulsion all resulted in an inferior kill.

A comparison is given in table 6 of the results obtained in dipping *Popillia* grubs, embedded in soil-balls and *not* embedded in soil, in solutions of various compounds. When, for instance, *Popillia* grubs *not* embedded in soil-balls were dipped in saturated solutions of carbon disulfide, thymol, mustard oil, etc., the grubs were dead in 30 minutes. When however the grubs were embedded in soil-balls and the soil-balls dipped in the same strength of the above solutions for 24 hours the grubs were unharmed.

The question naturally arises as to why this class of slightly soluble compounds, comprising carbon disulfide, thymol, mustard oil, etc., so effective in killing *Popillia* grubs when the latter are *not* embedded in soil, should so signally fail to kill the grubs when they *are* embedded in soil. The following

tests would seem to indicate the reason for this dissimilarity in results. In making these tests a solution was allowed to percolate through the soil-column of sandy loam contained in the drain pipe as shown in plate 2, figure 3. This soil-column was 10 cm. in diameter and 20 cm. in height, giving a total soil-volume of approximately 1,570 cc. The percolate was collected at the bottom of the soil-column by means of the funnel and beaker.

Using the above apparatus, a saturated solution of thymol was gradually poured onto the top of the soil-column and allowed to percolate through the soil, the liquid being collected as it drained away from the column into the beaker. The first 600 cc. of the percolate gave no taste or odor of thymol, the next 100 cc. contained a trace of thymol and the percolate gradually increased in concentration of thymol until 3800 cc. had been collected. At this point a sample of the percolate contained virtually the same concentration of thymol as the solution introduced into the top of the soil-column. Substantially the same results were obtained with individual saturated solutions of carbon disulfide, mustard oil and chloroform; in each case no trace of the solute could be detected in the percolate until several hundred cubic centimeters had drained through the column and the concentration of the substance in the percolate was not equal to the concentration of the saturated solution until several thousand cubic centimeters had drained through the column. *In other words the material in solution is largely "filtered out" by the soil until the latter becomes saturated, whereupon the solution percolates through unchanged.* The exact cause of this removal of solute by soils cannot be specifically stated but is due in all probability to one or all of three causes, namely, soil absorption, soil adsorption, and chemical action between certain of the soil constituents. The last named phenomenon may be responsible for the removal of *some* materials when their solutions are allowed to percolate through soils, but the scope of chemical action in the soil is not broad enough to account for the almost automatic removal of practically everything in solution which is allowed to percolate through soil. The phenomenon would seem to be physical rather than chemical and in all probability the removal of the solute may be attributed in large part to the adsorption of the material in solution by the moisture film enveloping the minute soil particles.<sup>3</sup>

<sup>3</sup> Detailed investigations by various soil physicists have shown that the individual moisture-films surrounding these minute soil particles exist or are held under comparatively tremendous force or pressure. Instances have been recorded of a pressure of 6,000 to 25,000 atmospheres. This pressure or compression results in a pronounced liquid density of the moisture-film, consequently there is a decidedly greater *quantity* of liquid present in the films than would be the case if it were under a pressure of only one atmosphere. Since there is more liquid present in the film it follows that this compressed liquid can dissolve or (absorb) more of a given solute than could the same *volume* of liquid under normal conditions of pressure, etc. Under these circumstances when a solution is introduced into the soil it percolates through the pore space where it comes in contact with the films surrounding the soil particles and these moisture films absorb the solute to the limit of their capacity.

This would seem to indicate that when solutions of soil insecticides are added to soil the material in solution is adsorbed up to a point governed by the capacity of the moisture films surrounding the soil particle. The experimental data also indicate that that portion of the toxic solute adsorbed by the soil particles is largely rendered *impotent* as far as its ability to act as a killing agent against insects in the soil is concerned. *Apparently the unadsorbed portion of the toxic solute is responsible for any mortality of soil insects resulting from the use of the solutions.* The quantity of solute remaining unadsorbed in the soil, when a given quantity of solution is added to a given quantity of soil, will depend upon the *initial* strength of the solution.

The limitations which this phenomenon of soil adsorption imposes upon the process of dipping soil-balls in solutions toxic to insects are evident when one considers the inter-action of the soil-ball and the dip. When a ball of earth wrapped in burlap is submerged in a liquid, the liquid penetrates into the soil-ball, largely displacing the air from the pore-space of the soil. *When the liquid has filled the soil pore-space, the movement of liquid into the soil-ball practically ceases.*

If the above liquid is a solution the adsorption of the solute by the soil particles goes on while the liquid is penetrating into the soil-ball and continues for some time after this penetration is complete and the liquid within and without the soil-ball is in a state of relative rest. It is obvious that the amount of the solute finally remaining unadsorbed in the liquid at rest within the soil-ball will depend absolutely on the initial concentration of the solution. Relatively dilute solutions of toxic compounds are largely adsorbed, leaving little unadsorbed material physically free to produce insect mortality. This explains the failure of saturated solutions of carbon disulfide, thymol, etc., to produce mortality of *Popillia* grubs embedded in soil-balls, although such solutions kill the grubs readily enough when the latter are not embedded in soil. *The saturated solutions of these materials are relatively dilute and the solute is largely adsorbed.*

Of the various compounds tested only two produced 100 per cent mortality of *Popillia* grubs when the latter were embedded in soil.<sup>4</sup> These two compounds were sodium sulfocarbonate and sodium ethyl xanthate. The positive results obtained with these two compounds are due to the fact that, unlike carbon disulfide, they are soluble in water in all proportions. By dipping *Popillia* grubs embedded in soil-balls in sufficiently concentrated

<sup>4</sup> The soil-balls used for these dipping tests contained 9 grubs (see page 50 for description of these balls). One grub was placed in the center of the ball and the remaining 8 distributed around the top, bottom and sides of the ball, within  $\frac{1}{2}$  inch of the surface. The grub in the center of the ball invariably escaped the action of the toxic gas when comparatively dilute dipping solutions were used, although one or more of the grubs located near the surface of the ball might succumb. It was only by using comparatively concentrated solutions that the grub in the center of the ball could be killed, in which case, as a matter of course, the grubs near the surface of the ball succumbed also.

solutions of these materials—for instance, 30 cc. of sodium sulfocarbonate to the gallon—an amount of the material sufficient to produce 100 per cent mortality of the grubs remains unadsorbed in the soil.<sup>5</sup>

The grubs in the soil are killed to some extent by contact with the sodium sulfocarbonate itself, but the efficiency of the material is due largely to the formation of carbon disulfide by the action of organic acids. The toxicity of sodium ethyl xanthate is entirely due to its decomposition into carbon disulfide by organic acids.

#### DOSAGE TESTS WITH BALLED CONIFEROUS PLANTS

Plate 1 illustrates the type and size of coniferous plants employed in the dosage tests outlined in table 7. The plants varied from 8 to 12 inches in height and were balled and wrapped as for shipment before they were dipped in the various solutions. In dipping, the ball of earth was completely submerged in the solution and allowed to remain undisturbed throughout the duration of the dipping period. Galvanized-iron tubs or buckets were used as containers for the solutions. On removal from the solution the plants were allowed to drain for 3 days in a room maintained at a temperature of 70°F. and were then planted in a small experimental nursery and kept under observation during the remainder of the season.

Table 7 gives the results of dipping coniferous plants in various solutions of three compounds, namely, sodium cyanide, sodium sulfocarbonate and sodium ethyl xanthate. Sodium cyanide in dilute solutions was extremely toxic to the plant, while the compound gave poor results against *Popillia* grubs embedded in soil. Sodium sulfocarbonate when used at the rate of 30 cc. per gallon of water, a dose toxic to *Popillia* grubs embedded in soil, shattered the roots of the plants and the tops were completely browned in from 10 to 15 days. The table indicates the results with smaller doses of this compound. In the case of trees treated with 10 cc. of sodium sulfocarbonate per gallon on May 20, the foliage was unimpaired but an examination of the root system 10 days after the treatment disclosed the fact that the root tips had been killed. Twenty days after treatment new root tips had formed and the tree resumed growth. However, during the season the tree made only one-half the growth of the check trees, indicating a slowing down as a result of the treatment with sodium sulfocarbonate.

Sodium ethyl xanthate, when used at a strength capable of killing *Popillia* grubs embedded in soil, damaged the tree in virtually the same manner as did the sodium sulfocarbonate.

<sup>5</sup> Various tests conducted during the course of the above experiments indicate that the larvae of *Popillia japonica* are more resistant to toxic compounds than are other similar soil-inhabiting larvae such as *Lachnosterna*. Sasser and Sanford (2) found that the larvae of *Popillia japonica* were the most difficult to kill of the various larvae subjected to the gas.



TABLE 7  
Results of dipping balled coniferous plants in various solutions toxic to *Popillia grubs*

DATE	MATERIAL EMPLOYED	CONCENTRATION OF SOLUTION	DURATION OF DIPPING	SOIL TEMPERATURE °F.	SOLUTION TEMPERATURE °F.	EFFECT OF SOLUTION ON PLANTS
May 5	Sodium cyanide	3.5 gm. per gal.	45 min.	44-70	90-78	Dead in 12 days*
May 2	Sodium cyanide	7.0 gm. per gal.	45 min.	44-70	90-78	Dead in 6 days
May 2	Water only (checks)	Check	45 min.	44-70	90-78	Made vigorous growth during season
May 20	Sodium sulfocarbonate	10.0 cc. per gal.	15 min.	64-70	74-72	Made one-half normal growth during season
May 20	Sodium sulfocarbonate	15.0 cc. per gal.	15 min.	64-71	75-73	No growth during season
May 20	Sodium sulfocarbonate	20.0 cc. per gal.	15 min.	64-71	75-73	1 dead in 20 days, 2 made no growth during season
May 20	Sodium sulfocarbonate	30.0 cc. per gal.	15 min.	64-73	76-75	Dead in 10 days
May 20	Water only (checks)	Check	15 min.	64-71	75-73	Made vigorous growth during season
July 6	Sodium sulfocarbonate	15.0 cc. per gal.	1 hr.	77	76	Dead in 20 days
July 6	Sodium sulfocarbonate	30.0 cc. per gal.	1 hr.	77	76	Dead in 15 days
July 6	Sodium sulfocarbonate	15.0 cc. per gal.	1 hr.	76	75	Trees badly shattered; will die
July 6	Sodium sulfocarbonate	20.0 cc. per gal.	1 hr.	71	75	Dead in 15 days
July 6	Sodium sulfocarbonate	20.0 cc. per gal.	1 hr.	77	76	Dead in 15 days
July 6	Sodium sulfocarbonate	25.0 cc. per gal.	1 hr.	77	76	Dead in 15 days
July 6	Sodium sulfocarbonate	25.0 cc. per gal.	1 hr.	71	75	Dead in 15 days
July 6	Sodium sulfocarbonate	30.0 cc. per gal.	1 hr.	77	76	Trees badly shattered; will die
July 6	Sodium ethyl xanthate	20.0 cc. per gal.	1 hr.	77	76	Trees badly shattered; will die
July 6	Sodium ethyl xanthate	20.0 cc. per gal.	1 hr.	77	76	Dead in 20 days
July 6	Sodium ethyl xanthate	40.0 cc. per gal.	1 hr.	77	76	Trees injured slightly
July 6	Sodium ethyl xanthate	40.0 cc. per gal.	1 hr.	77	76	Made vigorous growth during season
July 6	36 per cent acetic acid	20 cc. per gal.	1 hr.	77	76	Trees injured slightly
July 6	Water only (checks)	Check	1 hr.	77	76	Made vigorous growth during season

\* The conifers used for tests made May 20 were *Thuja serruana*, all remaining tests were made with *Retinospora Pisifera aurea*.



## SUMMARY

Dipping tests indicate that certain compounds in solution, capable of producing a gas insoluble or only slightly soluble in water, are toxic to *Popillia* grubs. These compounds may be divided into two classes:

I. Compounds slightly soluble in water, e.g., carbon disulfide, thymol, mustard oil, etc.

II. Compounds readily soluble in water such as sodium sulfocarbonate and sodium ethyl xanthate. These compounds in solution, on being decomposed by organic acids yield carbon disulfide, the active killing agent.

Saturated solutions of compounds in class I (about 1 to 1,000) readily kill *Popillia* grubs when the latter are removed from the soil and dipped in the solution for a definite period of time. However, when *Popillia* grubs are embedded in a soil-ball and the latter dipped in these solutions the grubs contained within the soil-ball remain unharmed. Soil adsorption, or in other words physical "locking up" of the compound in solution by the moisture film surrounding the minute soil particles, is the apparent reason for the failure of these relatively dilute solutions to function in soil. That portion of the compound adsorbed by the soil is apparently rendered impotent as far as its ability to produce grub mortality in the soil is concerned.

Compounds of class II when used in dilute solutions give results comparable to those obtained in the employment of compounds in class I. However, when compounds of class II are employed in relatively concentrated solutions, a quantity of the compound sufficient to produce 100 per cent mortality of *Popillia* grubs remains free in the soil after the soil particles have adsorbed the compound to the limit of their capacity.

The comparatively concentrated solutions of sodium sulfocarbonate and sodium ethyl xanthate, when used for the treatment of balled earth about the roots of coniferous plants for the control of Japanese beetle larvae, injure the plants to an extent which prohibits the use of these compounds in practice.

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## PLATE 1

FIG. 1. Small coniferous plant with ball of earth about the roots; the plant has just been taken from the nursery row.

FIG. 2. Same plant as in figure 1, but wrapped in burlap for shipment.



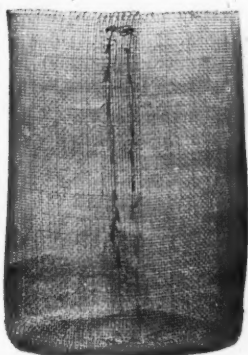
PLATE 2

FIG. 1. Wire cylinder 8 inches in diameter and 10 inches in height with wire bottom.

FIG. 2. Same cylinder as in figure 1, covered with double thickness of muslin; employed as container for artificial soil-ball.

FIG. 3. Apparatus employed in studying the percolation of toxic solutions through soil.

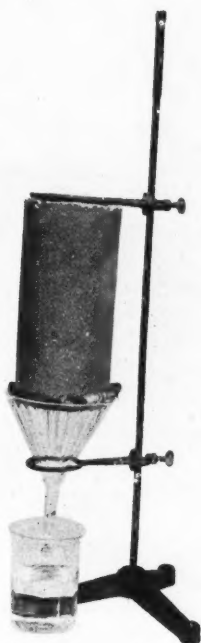
FIG. 4. Small wire cage, with removable cork stoppers, used as containers for *Popillia* grubs when embedded in soil-balls.



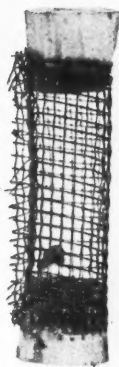
1



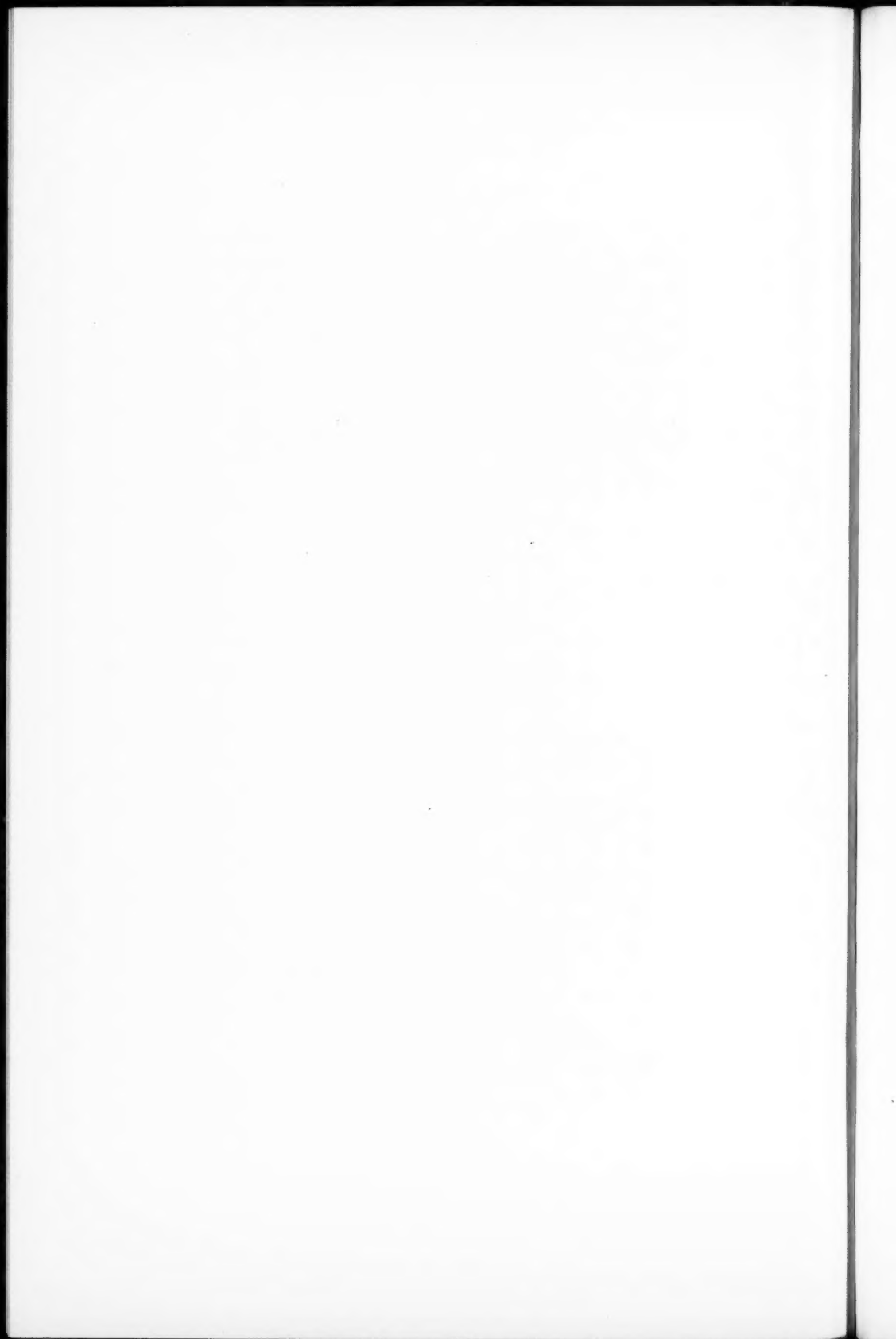
2



3



4



## EXPERIMENTS WITH HOT WATER IN THE TREATMENT OF BALLED EARTH ABOUT THE ROOTS OF PLANTS FOR THE CONTROL OF JAPANESE BEETLE LARVAE

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One of the experimental problems in connection with the control of the green Japanese beetle (*Popillia japonica* Newm.) consists in finding a method of killing *Popillia* grubs which may possibly occur in balled earth about the roots of coniferous and certain other ornamental plants or in the soil in which potted plants are grown. Such a method is needed because there are several nurseries growing these classes of plants within the area in New Jersey and Pennsylvania infested by the beetle and the marketing of their product is regulated by state and federal quarantines. Among other experiments (1) carried out during 1920 on this phase of the Japanese beetle problem the writer tested hot water (100° to 130°F.) in order to determine its value as a practical control measure for the treatment of balled earth.

### NURSERY PRACTICE IN SHIPPING CONIFEROUS PLANTS

In order that coniferous plants, such as *Arbor vitae* spruces, pines, etc., may be successfully transplanted, it is essential that the tree be dug in such a way that a sufficient proportion of the roots and rootlets are not disarranged in the soil or exposed to the drying action of the atmosphere. In order to secure a good soil-ball about the roots, nurserymen transplant conifers at frequent intervals in the nursery row. This frequent transplanting, with the accompanying root-pruning, results in a compact mass of fibrous roots in the immediate vicinity of the base of the tree. This compact mass of roots and soil, or the *soil-ball* as it is called, facilitates handling and insures successful transplanting. As an added precaution in shipping this class of stock the soil-ball is wrapped in burlap.

### BASIS OF THE EXPERIMENTAL WORK

As stated above, this class of nursery stock when ready for shipment is tightly wrapped in burlap bagging. Under these circumstances the plants can be dipped or soaked in water with impunity as far as mechanical injury is concerned, since only a very small proportion of the soil escapes through the bagging and the relative position of the roots within the soil-ball is undisturbed. On removal from the liquid the plants would be ready for shipment after being allowed to drain for a few hours.

## EXPERIMENTAL PROCEDURE

Aside from the few initial experiments the following steps were taken in testing hot water:

1. *Popillia* grubs were dipped in water at varying temperatures for varying periods of time in order to determine their resistance.
2. *Popillia* grubs, embedded in soil-balls, were subjected to the action of heated water.
3. Heated water was tested as to its action on the plant; small coniferous plants and azaleas, both with balled earth about their roots, were employed.

DIPPING TESTS WITH GRUBS OF *POPILLIA JAPONICA*

Tests were made in order to determine the degree of resistance of the grub when dipped in water at varying temperatures. The apparatus employed consisted of a small gas-burner, two galvanized-iron tubs, the smaller having about one-half the capacity of the larger, and a pair of long chemical Fahrenheit thermometers. The large tub was placed on the gas range and partially filled with water so that the small tub half-filled with water floated in the large tub. This arrangement is comparable to a water bath or double-boiler on a large scale.

On applying heat the water in the large tub slowly increased in temperature and also warmed up the water in the small tub. By adjusting the flow of gas it was possible to keep the water in the large tub at a fairly even temperature while the water in the small tub could be kept at a slightly lower and fairly constant temperature.

When the water in the small tub had remained at the desired temperature for half an hour, thereby indicating that the fluctuation in temperature was negligible from an experimental standpoint, the grubs, confined in small wire cages, were introduced into the water. After being subjected to the water for a definite period of time they were removed and placed on moist soil in tin salve-boxes for subsequent observation.

These tests indicate that, under the above experimental conditions, third-instar *Popillia* grubs have a certain resistance to water at various temperatures as follows.

*Popillia* grubs are killed by an exposure of 1 to 2 minutes in water at 130°F., 3 to 4 minutes in water at 125°F., and 6 to 8 minutes at 120°. One cannot bear the hand in water at these temperatures for any length of time.

The grubs are killed by an exposure of 12 minutes in water at 115°F. One can hardly bear the hand in water at this temperature.

The grubs are killed by an exposure of 45 minutes in water at 110°F. Water at this temperature is barely comfortable to the hand.

The grubs withstand an exposure of 2 hours in water at 105°. Water at this temperature is just comfortable to the hand.

*Popillia* grubs withstand an exposure of 24 hours in cold water (50°F.).



## EXPERIMENTS WITH POPILLIA GRUBS EMBEDDED IN SOIL

A soil-ball 8 inches in diameter was prepared from garden loam. During the process of manufacture 6 grubs, each in a small individual wire cage ( $1\frac{1}{2}$  inches long and  $\frac{1}{2}$  inch in diameter), were placed in the ball, two in the center and the remaining four within an inch of the surface. The ball was then wrapped in burlap and placed in water at the desired experimental temperature, the apparatus described above being employed.

Several tests at varying water temperatures were made of which the test represented graphically in figure 1 is representative. The curve expressed by the upper line represents the gradation in the temperature of the water from an initial temperature of  $110^{\circ}\text{F}$ . (the temperature when the soil-ball was introduced). The lower curve represents the gradation in temperature of the center of the soil-ball during its exposure to the heated water.

The soil-ball, at the moment of introduction into the water heated to  $110^{\circ}$ , had a temperature of  $51^{\circ}\text{F}$ . Five minutes afterward the temperature of the center of the ball had jumped to  $81^{\circ}\text{F}$ ., while the temperature of the water surrounding the ball had dropped to  $105^{\circ}\text{F}$ . The increase of  $30^{\circ}$  in the temperature of the soil-ball (measured at the center) is due to the inrush of the warm water into the air-spaces of the soil. The water surrounding the ball is cooled from  $110^{\circ}$  to  $105^{\circ}$  by imparting heat to the ball.

Under these circumstances, 5 minutes after the soil-ball had been placed in the water the temperature of the water had fallen to  $105^{\circ}$  and the temperature of the ball (*this included the water taken up by the ball*) had risen to  $81^{\circ}$ . An examination of the graph shows that during the next 65 minutes (the heat having remained constant) the temperature of the soil-ball gradually crept up to  $101^{\circ}\text{F}$ . while the water surrounding the ball remained constant at  $105^{\circ}$ .

During the next 75 minutes the soil-ball and surrounding water both increased in temperature and at the end of this period the surrounding water had attained its initial temperature of  $110^{\circ}$ . However, at this time the soil-ball still registered only  $106^{\circ}$  and 20 minutes elapsed before the center of the soil-ball registered  $110^{\circ}$ . During these 20 minutes it was necessary to decrease slightly the amount of heat in order to prevent the temperature of the water surrounding the ball from going above  $110^{\circ}$ .

It therefore required 160 minutes to bring the soil-ball up to the desired temperature of  $110^{\circ}$  and the ball was continued in the water at this temperature for an additional 45 minutes, at the end of which period it was removed and allowed to drain.

While draining, the surface of the ball cooled rapidly but the center persisted at a temperature of  $110^{\circ}$  for 15 minutes and then slowly cooled to room temperature as indicated in figure 1.

On the cooling of the soil-ball to room temperature the grubs were removed for examination. Tests conducted in the fashion described above gave the following results.

When a soil-ball containing *Popillia* grubs is placed in water at 110°, the ball brought up to a temperature of 110° and maintained at 110° for 45 minutes, the grubs all die. If the temperature is maintained in the same way at 105° only 66 per cent of the grubs succumb. At 100° only 33 per cent succumb and below this temperature they are not affected.

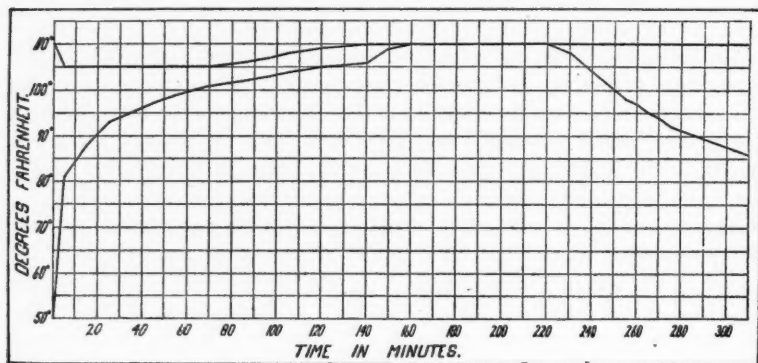


FIG. 1. RANGE AND VARIATION IN TEMPERATURE OF SOIL-BALL AND WATER BATH

#### TREATMENT OF BALLED-PLANTS WITH HEATED WATER

Two species of plants, which in practice are shipped with earth about their roots, were treated with heated water. In these tests the plants were placed in the water at the desired temperature, the ball of earth containing the roots allowed to warm up to that temperature and then maintained in the water at that temperature for 45 minutes. They were then removed from the water and allowed to drain for 2 days at room temperature before being planted in the nursery row. These treated plants were kept under observation during the remainder of the season.

#### *Results with Azalea amoena*

The balls of earth containing the roots of these plants were about 8 inches in diameter.

Plants treated as above at a temperature of 120°F. were checked decidedly in their growth during the season.

Plants treated at a temperature of 115°F. were appreciably checked in their growth during the season.

Plants (checks) treated at tap-water temperatures made normal growth during the season.

A few days after the treatment of these plants the roots were carefully examined for injury. No evidence of injury could be detected in roots large

enough to cross-section by hand but the subsequent growth of the plants during the season indicated that the small feeding rootlets were injured by the treatment.

#### *Results with coniferous plants*

The plants employed in these tests were *Retinospora Pisifera aurea*, a variety of *Arbor vitae*. The ball of earth containing the roots was about 8 inches in diameter.

Plants treated at a temperature of 110°F. were badly injured. Some died and in others half the foliage was dead at the end of the season. The main roots were injured severely. Cells throughout the entire cross-section of these roots were killed.

Plants treated at a temperature of 105°F. were decidedly affected. The roots were injured and the foliage made practically no growth during the season.

Plants treated at a temperature of 100°F. were affected to some extent by the treatment. The small rootlets were killed and the top did not begin to send out new foliage until late in the season.

#### ANALYSIS OF RESULTS

The experimental data outlined above clearly indicate that the roots of plants are less capable of withstanding exposure to heated water than are the larvae of *Popillia japonica*. When roots are exposed to water at the minimum temperature necessary to insure the mortality of the grubs the roots are literally "cooked" with the result that the plant succumbs or its subsequent growth is at least decidedly checked.

Aside from the question of injury to the plant the treatment would not seem to lend itself to actual practice due to the slowness of the method and difficulty of controlling the temperature of the water. This last fact is evident when one considers the problem of heat radiation from the water-container resulting in a variation of several degrees in temperature between the bottom, sides and center of the mass of water. The slowness of the method is evident when one considers that it requires more than 3 hours to treat a small ball of earth.

#### SUMMARY AND CONCLUSIONS

The third-instar grubs of *Popillia japonica* withstand an exposure of 2 hours in water heated to a temperature of 105°F.; they succumb to an exposure of 45 minutes in water at a temperature of 110°F., to an exposure of 12 minutes at 115°F., to an exposure of 6 to 8 minutes at 120°F., to an exposure of 3 to 4 minutes at 125° and to an exposure of 1 to 2 minutes at 130°F.

When a bath of heated water (minimum temperature 110°F.) is used as a method of killing *Popillia* grubs in the balled-earth about the roots of plants

the soil-ball must be immersed until brought up to a temperature of 110°F. and remain immersed at that temperature for a period of 45 minutes. The process of warming up the soil is necessarily slow because it is not safe to allow the temperature of the water-bath to exceed 110°F. at any time. It requires 3 hours and 25 minutes to treat a soil-ball 8 inches in diameter by this method. Under the circumstances, and aside from the consideration of injury to the plant, the method is too slow to be utilized to any extent in practice.

The results indicate that plants vary considerably in their resistance to immersion in heated water for various periods of time at various temperatures, but that even the more resistant plants are affected and checked in their subsequent growth.

#### REFERENCE

- (1) LEACH, B. R., AND THOMSON, J. W. 1921 Experiments in the treatment of balled earth about the roots of coniferous plants for the control of Japanese beetle larvae. *In* Soil Sci., v. 12, no. 1, p. 43-61.

# THE RELATION OF THE HYDROGEN-ION CONCENTRATION OF NUTRIENT SOLUTIONS TO GROWTH AND CHLOROSIS OF WHEAT PLANTS

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## INTRODUCTION

In a previous paper (4) the writers published the results of an investigation of the hydrogen-ion concentration of the three-salt nutrient solutions recommended (3) by a special committee of the Division of Biology and Agriculture of the National Research Council. These results indicated that none of the differences in the growth rate of wheat plants in sand cultures with any one type of solution could be attributed to variations in the hydrogen-ion concentration of the nutrient solutions. This, however, does not preclude the possibility that the relative merit of the six types of solutions is influenced to some extent by their respective ranges of H-ion concentrations. Table 1, summarized from the previous paper (4), shows the magnitude of the pH ranges.

TABLE 1  
*Composition and pH ranges of the three-salt solutions*

TYPE	CALCIUM SALT	POTASSIUM SALT	MAGNESIUM SALT	pH RANGE
I	$\text{Ca}(\text{NO}_3)_2$	$\text{KH}_2\text{PO}_4$	$\text{MgSO}_4$	4.4-4.8
II	$\text{Ca}(\text{NO}_3)_2$	$\text{K}_2\text{SO}_4$	$\text{Mg}(\text{H}_2\text{PO}_4)_2$	3.5-3.8
III	$\text{Ca}(\text{H}_2\text{PO}_4)_2$	$\text{KNO}_3$	$\text{MgSO}_4$	3.5-4.1
IV	$\text{Ca}(\text{H}_2\text{PO}_4)_2$	$\text{K}_2\text{SO}_4$	$\text{Mg}(\text{NO}_3)_2$	3.5-4.1
V	$\text{CaSO}_4$	$\text{KNO}_3$	$\text{Mg}(\text{H}_2\text{PO}_4)_2$	3.5-4.3
VI	$\text{CaSO}_4$	$\text{KH}_2\text{PO}_4$	$\text{Mg}(\text{NO}_3)_2$	4.8-5.3

The types containing potassium phosphate ( $\text{KH}_2\text{PO}_4$ ) have a lower H-ion concentration than those containing either magnesium phosphate, ( $\text{Mg}(\text{H}_2\text{PO}_4)_2$ ) or calcium phosphate, ( $\text{Ca}(\text{H}_2\text{PO}_4)_2$ ). It may be added that, in general, within any one type, the H-ion concentration of the solution is a function of the volume-molecular proportion of the di-hydrogenphosphate salt present. These considerations, together with the existence of only a very limited amount of work bearing on the relation of the H-ion concentration of nutrient solutions to the growth of the higher plants, suggested to the authors the desirability of undertaking the work which is described in this paper.

## PLAN OF THE EXPERIMENT

Wheat was grown in sand (3) cultures for a period of 2 months. The nutrient solutions were changed twice a week. Four different solutions were used as the basis of this experiment. Each basic solution was modified by the additions of 0.025 *M* potassium sulfate, 0.025 *M* sulfuric acid or 0.05 *M* potassium hydroxide<sup>1</sup> at the rate of 20 cc. to each liter of nutrient solution. Potassium hydroxide and sulfuric acid were added to vary the H-ion concentration while potassium sulfate was added to check up the effect of the extra potassium or sulfur added in the form of potassium hydroxide or sulfuric acid. Each basic solution was modified in this manner so as to have three distinctly different pH values without materially altering the relative concentration of the six essential ions. The pH values of the solutions thus produced varied from 3.06 to 7.00.

The basic solutions used were I R<sub>3</sub>S<sub>3</sub>, III R<sub>3</sub>S<sub>3</sub>, as described by a special committee of the National Research Council (3), and two concentrations of a special solution so made up as to contain the essential ions in approximately the same proportion as they are found in a normal wheat plant. The stock solution from which this solution was made had the following composition:

	grams per liter
Calcium phosphate (Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub> ).....	4.10
Calcium nitrate (Ca(NO <sub>3</sub> ) <sub>2</sub> ).....	18.00
Potassium nitrate (KNO <sub>3</sub> ).....	7.80
Magnesium nitrate (Mg(NO <sub>3</sub> ) <sub>2</sub> ).....	0.20
Magnesium sulfate (MgSO <sub>4</sub> ).....	0.75
Sodium chloride (NaCl).....	0.10

This stock solution was diluted 1 to 12 and 1 to 24. When diluted 1 to 12 a nutrient solution was produced having an osmotic pressure of 1.06 atmospheres. The above solutions, for convenience, will be referred to as I R<sub>3</sub>S<sub>3</sub>, III R<sub>3</sub>S<sub>3</sub>, Special 1-12, and Special 1-24.

## DATA AND DISCUSSION

The data upon which the discussion is based are given in table 2. This table also furnishes the basis for the curves shown in figure 1. The outstanding feature of the results is the very marked influence of the H-ion concentration upon growth and chlorosis. The physico-chemical properties of the solutions themselves are very interesting and will be given consideration under a separate heading.

<sup>1</sup> In terms of normality 0.025 *M* sulfuric acid and 0.05 *M* potassium hydroxide are equivalent, each having a reacting power equivalent to 0.05 *N* solutions.

TABLE 2

*Weight of tops and appearance of wheat plants grown in sand cultures with nutrient solutions having different hydrogen-ion concentrations*

CULTURE NUMBER	BASIC SOLUTIONS	20 CC. ADDITIONS TO EACH LITER OF BASIC SOLUTION	HYDROGEN-ION CONCENTRATIONS OF NUTRIENT SOLUTION	GREEN WEIGHT OF TOPS (5 PLANTS)	AVERAGE GREEN WEIGHT	APPEARANCE OF PLANTS FROM 2 WEEKS AFTER PLANTING TO HARVEST
			<i>pH</i>	<i>gm.</i>	<i>gm.</i>	
1 2	I $R_2S_3$	None	4.60 {	14.0 13.8	13.9	Very chlorotic
3 4		0.025 M $K_2SO_4$	4.68 {	18.0 14.8		
5 6		0.025 M $H_2SO_4$	3.26 {	36.0 31.2	33.6	Green
7 8		0.05 M KOH	5.83 {	7.5 9.1		
9 10	III $R_2S_3$	None	3.53 {	24.7 25.6	25.2	Dark green
11 12		0.025 M $K_2SO_4$	3.56 {	23.4 23.0	23.2	Dark green
13 14		0.025 M $H_2SO_4$	3.11 {	27.3 25.0	26.2	Very dark green
15 16		0.05 M KOH	4.84 {	15.4 14.6	15.2	Very chlorotic
17 18	Special 1-12	None	4.02 {	26.0 22.6	24.3	Chlorotic
19 20		0.025 M $K_2SO_4$	4.06 {	22.6 30.7	26.2	Chlorotic
21 22		0.025 M $H_2SO_4$	3.06 {	41.3 37.4	39.9	Very dark green
23 24		0.05 M KOH	6.27 {	15.2 17.4	16.3	Very chlorotic
25 26	Special 1-24	None	4.33 {	21.2 26.2	23.7	Very chlorotic
27 28		0.025 M $K_2SO_4$	4.37 {	20.0 18.2	19.1	Very chlorotic
29 30		0.025 M $H_2SO_4$	3.14 {	49.0 48.8	48.9	Very dark green
31 32		0.05 M KOH	7.00 {	17.0 19.1	18.1	Very chlorotic



## GROWTH

The curves given in figure 1 show the relation between the pH values and the green weight of tops for wheat plants growing in sand cultures and supplied with the different nutrient solutions. In considering the rôle of the H-ion concentration and its relation to growth, the relative merit of the basic nutrient solutions must be taken into consideration. An examination of the curves of figure 1 shows that solutions "Special 1-12 and 1-24" when adjusted to the proper H-ion concentration are distinctly superior to I  $R_3S_3$  and III  $R_3S_3$ . It is impossible, therefore, to plot all pH values and all green weights of tops against each other regardless of the basic nutrient solutions used. It will be

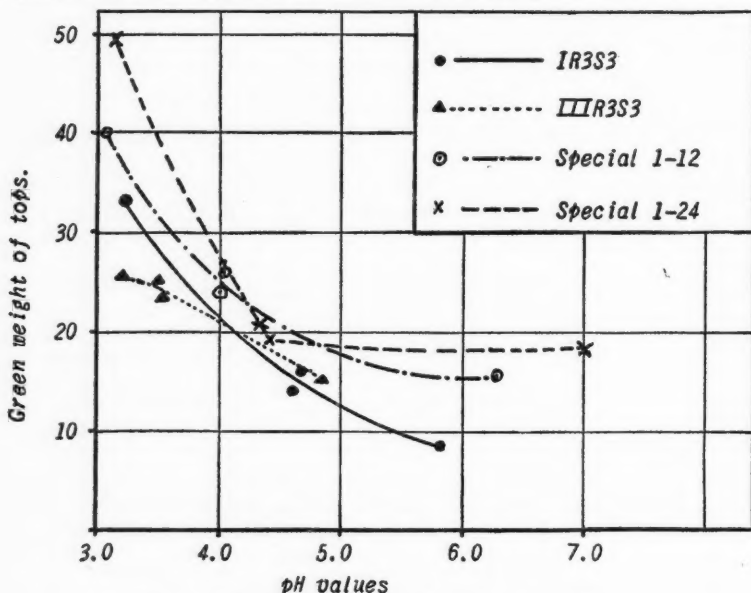


FIG. 1. GREEN WEIGHTS PLOTTED AGAINST pH VALUES

seen from table 2 that all solutions, except III  $R_3S_3$  were greatly improved with respect to the green weight of tops when 0.025  $M$  sulfuric acid was added at the rate of 20 cc. per liter of nutrient solution. The addition of the same amount of 0.05  $M$  potassium hydroxide depressed the growth to a marked extent. Since an equivalent amount of 0.025  $M$  potassium sulfate produced no noticeable effect, all significant variations in growth can be attributed to the differences in the H-ion concentration. The addition of sulfuric acid to III  $R_3S_3$  did not increase the growth rate. This solution is not only quite acid to begin with, but as will be pointed out later, is also highly buffered. It is not surprising, therefore, that this solution behaves somewhat differently

from the other three solutions under consideration. From the curves of figure 1 it will be seen that in every case the highest green weight of tops was secured from the solutions whose pH values were less than 4.

Hoagland (2) reports that solutions having a pH value of 5.0 were not injurious to barley plants. Our results also favor a much higher H-ion concentration for optimum growth of wheat plants than does the work of Duggar (1) and that of Salter and McIlvaine (6). It should be mentioned, however, that these workers employed solution cultures, whereas our plants were grown in sand cultures.

#### CHLOROSIS

Regardless of the basic nutrient solution used, the H-ion concentration was closely correlated with the presence or the absence of chlorosis in the plants of our cultures. The chlorotic condition varied from a slight tendency toward a light green color to a practically complete absence of chlorophyll.

The seed was placed on the germinator October 23 and 5 days later the seedlings were transferred to the culture pots. About November 5 slight chlorosis was evident in some of the cultures but it was impossible to correlate this chlorotic condition with the H-ion concentration until about 10 days later. From November 15 until the plants were harvested, all solutions having pH values of 3.06 to 3.56 produced normal green plants, while those having pH values of 4.02 to 7.00 produced chlorotic plants.

The physiological rôle of the H-ion concentration is not understood. The H-ion concentration may, of course, have a direct influence upon metabolism, or it may affect the cell permeability or it may have a direct effect upon the condition of the nutrient solution itself. The appearance of the etiolated plants together with the fact that no iron was employed in these culture solutions, except that contained in the sand, at once suggested iron chlorosis.

There are a number of different forms of chlorosis, the most common of which is usually attributed to the lack of available iron. In the field this condition is supposed to be caused by a lack of available iron due to the presence of excessive amounts of lime or other bases. The reaction of the soil solution may render the iron insoluble and thus cause it to become unavailable to the plant in sufficient amounts, or it may have a direct effect upon the metabolism of the plant itself, thus rendering immobile such iron as may actually get into the plant. Although we have no direct positive evidence that the chlorotic condition of our culture plants was due to faulty iron absorption or metabolism, a number of very interesting facts may be mentioned in connection with the iron situation.

In common with many other workers employing sand cultures, we have always regarded the iron contained in the sand as being available in sufficiently large amounts to supply the needs of our plants. There is a strong possibility that this is not true for all nutrient solutions. In a recent publication (5) Patten and Mains have shown that when sodium hydroxide or ammonium

hydroxide is added to a dilute solution of ferric chloride, ferric hydrate ( $\text{Fe}(\text{OH})_3$ ) is precipitated, if the resulting mixture has a pH value higher than 3.5. In their solutions a faint cloudiness was apparent at pH = 3.5, which increased to a fine precipitate at pH = 5.5, becoming very heavy at pH = 6.0. Nutrient solutions which were in contact with our growing plants from December 6 to 9 were tested for iron by means of potassium sulfocyanate, with the result that only cultures 13, 14, 21, 22, 29 and 30 gave a positive reaction for iron. It will be noticed that all of these nutrient solutions have a pH value between 3.06 and 3.14.

From December 14 until the time of harvesting, cultures 28 and 32 were given ferric nitrate ( $\text{Fe}(\text{NO}_3)_3$ ) at each feeding at the rate of 10 parts of iron per million of the culture solution. Culture 26 was given the regular amounts (20 cc.) of 0.025 *M* sulfuric acid. The added iron produced no apparent effect whatever. It will be noticed however that the cultures to which iron was added had pH values of 4.37 and 7.00. These pH values are such that one would expect that the iron would be thrown out of solution as ferric hydrate ( $\text{Fe}(\text{OH})_3$ ) and thus made unavailable to the plants. Culture 26, to which 20 cc. of 0.025 *M* sulfuric acid was added soon began to show a decided improvement over no. 25, its check, to which no additions of acid were made. At the time of harvest, culture 26 was normal in color while no. 25 retained its chlorotic condition to the end of the growth period. This again serves to emphasize the importance of the H-ion concentration, although it does not throw any light upon the effect of the reaction on the physiological functioning of the solution.

#### THE BUFFER ACTION OF THE NUTRIENT SOLUTIONS

In order to study the buffer properties of the nutrient solutions, the hydrogen-ion concentrations of the original basic solutions were determined before and after the 10-cc. and 20-cc. additions of the modifying solutions were made. All of the plant cultures, however, were grown in the original solutions and in solutions modified by the additions of 20-cc. portions of potassium sulfate, sulfuric acid or potassium hydroxide per liter of the nutrient solution.

In table 3 are recorded the effects of these additions upon the hydrogen-ion concentration of the nutrient solution. The buffer properties of the four basic solutions are brought out in the titration curves of figure 2. These curves, within the ranges covered, may be made use of in the following ways:

1. To determine the pH value at which a given solution is the most highly buffered, and conversely, the reaction at which a solution is most easily subject to changes in pH values.

2. To determine the amount of sulfuric acid or of potassium hydroxide that must be added to the basic solutions to secure any desired pH value. This type of information concerning nutrient solutions is very important for several reasons. It makes it possible systematically to prepare solutions of desired H-ion concentrations and to predict something as to the permanency of the

pH value of a given solution when exposed to plants for any length of time, since the highly buffered solutions are more resistant to change than those that exhibit but little buffer action. It is, of course, desirable to use nutrient

TABLE 3

*The effect upon the hydrogen-ion concentration of the additions of different amounts of acid and alkali to basic culture solutions*

BASIC SOLUTIONS	ADDITIONS TO BASIC SOLUTIONS	HYDROGEN-ION CONCENTRATIONS	
		After additions of 10 cc.	After additions of 20 cc.
		pH	pH
I R <sub>3</sub> S <sub>8</sub> .....	None	4.60	4.60
	0.025 M K <sub>2</sub> SO <sub>4</sub>	4.63	4.68
	0.025 M H <sub>2</sub> SO <sub>4</sub>	3.60	3.26
	0.05 M KOH	5.56	5.83
III R <sub>3</sub> S <sub>8</sub> .....	None	3.53	3.53
	0.025 M K <sub>2</sub> SO <sub>4</sub>	3.55	3.56
	0.025 M H <sub>2</sub> SO <sub>4</sub>	3.28	3.11
	0.05 M KOH	3.94	4.84
Special 1-12.....	None	4.02	4.02
	0.025 M K <sub>2</sub> SO <sub>4</sub>	4.06	4.06
	0.025 M H <sub>2</sub> SO <sub>4</sub>	3.32	3.06
	0.05 M KOH	5.88	6.27
Special 1-24.....	None	4.33	4.33
	0.025 M K <sub>2</sub> SO <sub>4</sub>	4.37	4.37
	0.025 M H <sub>2</sub> SO <sub>4</sub>	3.42	3.14
	0.05 M KOH	6.42	7.00

TABLE 4

*Magnitude of the change in pH values, produced by the addition of sulfuric acid and of potassium hydroxide to basic solutions*

BASIC NUTRIENT SOLUTION	pH VALUES OF ORIGINAL SOLUTIONS	CHANGE IN pH VALUES ON ADDITION OF			
		0.025 M H <sub>2</sub> SO <sub>4</sub>		0.05 M KOH	
		10 cc.	20 cc.	10 cc.	20 cc.
I R <sub>3</sub> S <sub>8</sub> .....	4.60	1.00	1.34	0.96	1.23
III R <sub>3</sub> S <sub>8</sub> .....	3.53	0.25	0.42	0.41	1.31
Special 1-12.....	4.02	0.70	0.96	1.86	2.25
Special 1-24.....	4.33	0.91	1.19	2.09	2.67

solutions whose reaction remains reasonably constant and thus avoid too frequent renewals of the solutions.

The data of table 3 are summarized in table 4 to show the magnitude of the changes in the pH values that are produced by certain additions to the basic nutrient solutions.

This table, in a general way, brings out the relative ease with which hydrogen-ion concentration of any solution may be changed by the addition of various increments of sulfuric acid or of potassium hydroxide.

Conversely, the same data will be found useful in the study of changes in the pH values of nutrient solutions after contact with plant roots. This information, of course, can be obtained directly from the titration curves. These suggestions are in agreement with the work of Salter and McIlvaine (6).

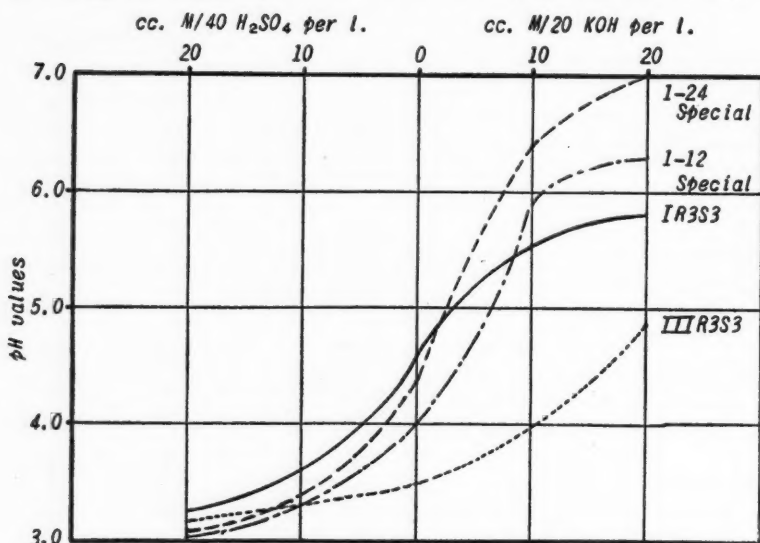


FIG. 2. TITRATION CURVES FOR BASIC SOLUTIONS USED<sup>2</sup>

#### SUMMARY

1. Wheat plants were grown for 2 months in four different basic nutrient solutions, each of which was modified in such a way as to have three distinctly different pH values without materially altering the solutions with respect to the concentration of the six essential ions.

2. The H-ion concentration not only exerted a very marked influence upon the growth rate but also was an important factor in the control of chlorosis.

3. Although the exact rôle of the H-ion concentration is not understood, the appearance of the chlorotic plants and some indirect evidence would indicate that our plants grown in solutions having pH values ranging from 4.02 to 7.0 were suffering from the lack of available iron or from faulty metabolism resulting from the immobility of the iron within the plants.

4. The data presented emphasize the importance of the proper control of the H-ion concentration in all work of this kind.

<sup>2</sup> The terms M/40 H<sub>2</sub>SO<sub>4</sub> and M/20 KOH that appear in figure 2 are equivalent to the expressions 0.025 M H<sub>2</sub>SO<sub>4</sub> and 0.05 M KOH, respectively, as given in the text.

## REFERENCES

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